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TECHNICAL REPORT AFML-TR-66-75

NEW ABLATIVE PLASTICS AND COMPOSITES, THEIR FORMULATION AND PROCESSING

APRIL 1966

AEROSPACE GROUP
HUGHES
HUGHES AIRCRAFT COMPANY
CULVER CITY, CALIFORNIA

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(6) NEW ABLATIVE PLASTICS AND COMPOSITES, THEIR FORMULATION AND PROCESSING

(10) B. G. Kimmel and G. Schwartz
~~Hughes Aircraft Company~~

(9) Summary rept. Feb 65 – Feb 66

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Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by Hughes Aircraft Company, Culver City, California, under USAF Contract No. AF33(615)-2418. This contract was initiated under project No. 7340, "Non-Metallic and Composite Materials", Task No. 734001, "Thermally Protective Plastics and Composites". The work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, Research and Technology Division. Mr. P. F. Pirrung acted as project engineer.

This report covers work from February 1965 to February 1966.

Previous work on this contract was performed under USAF Contract No. AF33(657) 8621 and will be found in ASD TDR 63-568, Part I, ML TDR 64-222 and AFML-TR-65-94.

Manuscript released by the authors, March 1966 for publication as an RTD Technical Report.

This technical report has been reviewed and is approved.

R. G. SPAIN, Acting Chief
Plastics and Composites Branch
Nonmetallic Materials Division
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Research and Technology Division

ABSTRACT

Precise processing techniques were used in preparing new ablative plastics composites. This research involved the use of novel heat-resistant resins such as:

- branched, cross-linked polyphenylenes
- para-polyphenylenes
- amide-blocked polybenzimidazole
- phosphonitrilic-modified phenolic
- carborane
- polyimide

Novel reinforcements included:

- boron fibers
- boron nitride fibers
- fused silica fabric coated with pyrolytic graphite
- polybenzimidazole fibers
- rayon-silica fabric
- carbon-silica fabric
- sapphire wool fibers
- silicon carbide fibers

Resin impregnation techniques used in preparing research specimens included spatula coating, dip coating, machine coating in a laboratory treater, Buchner funnel impregnation and dry powder layup.

Research specimens of controlled composition were prepared and submitted to the Air Force Materials Laboratory for hyperthermal evaluation, as follows:

- pellet specimens, 3/4-inch diameter by 1/2-inch long.
- rocket nozzle assemblies ASD No. 4
- hot gas flow specimens of several types
- laminates, 6 x 6 x 1/8 inch
- laminated squares, 2 x 2 x 1/2 inch
- supersonic pipe specimens, 1.675-inch diameter by 1-1/2-inches long

CONTENTS

TABLES

INTRODUCTION

New polymeric materials and reinforcements have been developed in government and industry research programs. Many of these materials offer considerable promise for use in high-performance ablative plastics.

The program objectives are to select promising ablative materials for further study and develop suitable fabrication procedures for preparing ablative composites containing these new materials.

The work done during this 12-month period of the program consisted mainly of the continued use of precise processing techniques in fabricating research specimens of closely controlled composition. Specimens were produced consistently with a resin content within a ±2-percent range. In all experiments, all pertinent processing information and data were recorded to allow later duplication of any test specimens required for further tests. These processing data can be used in scaling up the processes if required.

Specimens prepared under this contract have been forwarded to the Nonmetallic Materials Division, Air Force Materials Laboratory, for subsequent hyperthermal evaluation.

Newly developed resins and reinforcements, which are becoming available in research quantities, were used to fabricate ablative composite specimens. These specimens will be subsequently characterized for possible use in high speed entry and rocket exhaust environments. Materials intended for potential entry environments will be characterized with an air arc heater. This research is being performed under AF 33(615)1937 with the Avco Corporation, RAD. Rocket nozzle specimens will be characterized using a liquid propellant motor or a solid propellant motor simulator under Contract AF 33(615)1632 with the Aeronutronic Division, Philco Corporation. Hot gas flow specimens will be characterized with a solid propellant motor, under contract AF 33(615)1631 with the Atlantic Research Corporation.

SUMMARY

Precise formulation and processing techniques were used in the preparation of ablative composites of controlled composition containing new polymeric materials and reinforcements.

Formulating, molding, and postcuring conditions were varied, as required, to produce test specimens of high quality from a wide range of resins and reinforcements. New resins investigated included:

- branched, cross-linked polyphenylenes
- para-polyphenylenes
- amide-blocked polybenzimidazole
- phosphonitrilic-modified phenolic
- carborane
- polyimide

Novel reinforcements included:

- boron fibers
- boron nitride fibers
- fused silica fabric coated with pyrolytic graphite
- polybenzimidazole fibers
- rayon-silica fabric
- carbon-silica fabric
- sapphire wool fibers
- silicon carbide fibers

In addition, a large quantity of research specimens were fabricated using standard resins such as phenolics or epoxy novolaks and standard reinforcements such as Refrasil, carbon cloth, or graphite cloth. In many cases, a standard reinforcement was combined with a new resin while a new reinforcement was combined with one of the standard resins.

During the period covered by this report, the following specimens were prepared and shipped to Air Force Materials Laboratory.

- 77 hot gas flow specimens
- 1 laminate
- 16 laminated squares
- 90 pellet specimens
- 40 ASD No. 4 rocket nozzle assemblies
- 32 supersonic pipe specimens

GENERAL SPECIMEN PREPARATION PROCEDURES

GENERAL DISCUSSION

Precise formulation and processing techniques were developed and applied in the fabrication of ablative composites containing new polymeric materials and reinforcements. Six main types of test specimens were prepared and submitted under this program:

- Hot gas flow

  Type A - 3.333 x 1.750 x 0.502 ±0.002 inch (each ply 1.750 x 0.502 inch)

  Type B - 3.333 x 1.750 x 0.502 ±0.002 inch (each ply 3.333 x 1.750 inch)

  Type D - 3.333 x 2.000 x 0.502 ±0.002 inch (each ply 3.333 x 2.000 inch)

  Type E - 3.333 x 1.750 x 0.502 ±0.002 inch (molded from 3/8-inch squares of impregnated reinforcement)
- Laminates - 6 x 6 x 1/8 inch
- Laminated squares - 2.000 x 2.000 x 0.502 ±0.002 inch
- Pellet specimens - 0.750 inch in diameter x 0.502 ±0.002 inch long
- Rocket nozzle assemblies - ASD No. 4
- Supersonic pipe specimens - 1.675 inch diameter x 1.5 inch minimum

A complete description of all test specimens fabricated and delivered during the period covered by this report is given in the fifteen tables in the Appendix.

Tables 1, 2, 3, 4, 5 and 6 give density, Barcol hardness, composition and a brief description of hot gas flow specimens, laminates, laminated squares, pellet specimens, rocket nozzles and supersonic pipe specimens.

Table 7 lists all specimens by increasing test specimen data sheet number. It also gives the specimen type, material codes, and other information such as the date requested and shipped.

Table 8 lists all test specimens according to type of reinforcement. Table 9 lists all test specimens according to type of resin.

Tables 10, 11, 12, 13, 14 and 15 give the fabrication details for hot gas flow specimens, laminates, laminated squares, pellet specimens, nozzles and miscellaneous moldings.

Table 16 lists Material Sources for resins, reinforcements and filler used.

The composition of the test specimens was maintained in almost all cases within the range of ±2 weight-percent of the required nominal composition. This was done by carefully controlling each step of the fabrication process from the initial coating of the reinforcement to the final postcure of the molded or laminated composite. Past experience was used in making allowance for the weight loss (change in composition) of the coated reinforcement which takes place upon drying, B-staging, curing and postcuring.

All reinforcements except glass and Refrasil were oven-dried two to three hours at 240°F prior to coating with resin. All subsequent calculations were based on this dry reinforcement weight. Carbon and graphite cloth have been found to lose as much as 10 weight-percent on drying.

TYPES OF IMPREGNATION

Several methods of impregnating the reinforcements were used:

- Spatula coating
- Dip coating
- Soaking
- Dry powder layup
- Machine coating in a laboratory coater

Spatula Coating

This method of impregnation was only on cloth. Fabric is cut to a size sufficient to allow the blanking or cutting out of the proper number of plies for the molding. The dry cloth is weighed and laid out on a piece of cellophane. The proper amount of resin is weighed out and thinned, if necessary, to coating consistency. The resin is poured over the fabric and uniformly distributed over the cloth with a spatula. The impregnated material is dried on the cellophane for 15 to 20 minutes, then hung up to dry for about one hour at room temperature. After drying at 160°F for 20 to 60 minutes, the cloth is weighed and the resin content calculated. Excess resin is removed by wiping the surface with a paper tissue soaked in thinner. However, if additional resin is needed, it is added to the backside of the cloth and uniformly distributed by a spatula. When the desired resin content is reached, the fabric is "B" staged to form a prepreg. The final resin content is then calculated from the final coated weight.

Dip Coating

This method of impregnation is used only on cloth. A weighed piece of dry cloth is passed through a small dipping tray repeatedly

until the required amount of resin is obtained. The cloth is allowed to dry after every fourth dip when a large number of dips are required. The drying time, from 5 to 30 minutes, depends on the resin system. Dip coating is usually used in place of spatula coating under the following circumstances:

- With solutions containing small percentages of resin solids
- With viscous resin solutions with large amounts of thinner added to obtain satisfactory coating properties
- When coating carbon or graphite cloth with a solution containing a high percentage of resin solids. These types of cloth tend to powder when spatula coated with a solution with a high solids content

When the correct resin content is obtained, the cloth is "B" staged and the final resin content is calculated as indicated above.

Soaking

This method is used with yarns, filaments, or fibers which wet readily. The dry material is placed in a beaker and a thinned resin solution containing a weighted amount of resin solids is added. The reinforcement is allowed to soak for 60 minutes in air before the excess solvent is removed by evaporation under vacuum. After drying in an oven at 160°F for 60 minutes, the resin content of impregnated reinforcement is calculated from the increase in weight.

The resin content of the prepreg is increased or decreased when required, by pouring additional resin or solvent over the material or filtering off the excess. The material is "B" staged after obtaining the proper resin content.

A slight modification of this method is referred to as Buchner funnel impregnation. The dry material is placed on a piece of filter paper (Whatman's No. 4 or equivalent) in a Buchner funnel. A thinned resin solution is poured into the funnel and the material soaked for five minutes. Excess resin is then removed by vacuum filtration.

Dry Powder Layup

Cloth cannot be impregnated when the resin used is a dry insoluble powder. Specimens are prepared by sprinkling resin between plies of reinforcement. First, pieces of cloth are blanked into plies and when required are dried in an oven at 300°F for one hour. A calculated amount of resin is sprinkled between plies with each addition of resin and cloth being weighed on an analytical balance. The resin and reinforcement are weighed into a preform holder and transferred into the cold mold prior to molding.

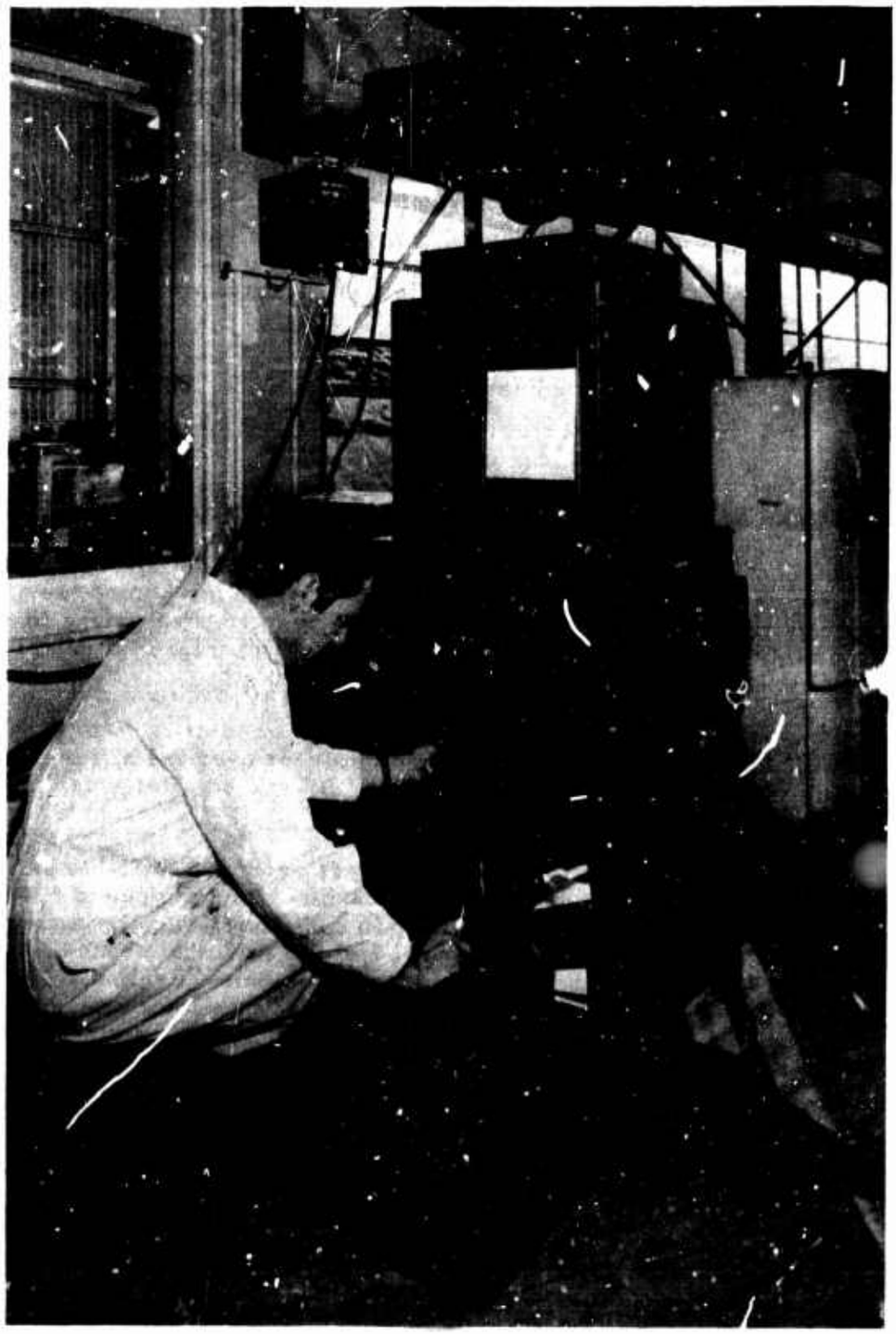


Figure 1. Laboratory coater.

Machine Coating

The laboratory coater is used to coat relatively large amounts (10 to 100 square feet) of fabric (Figure 1). Besides saving time over spatula or dip coating, this procedure results in a more uniform prepreg. The resin varnish, thinned to coating consistency with a suitable solvent, is poured into the coater diptank. A roll of dried, weighed cloth with a maximum width of 12 inches is placed in the coater. The coater speed can be varied from almost zero to 28 inches per minute. The total travel distance inside the coater is 59 inches. The temperature inside the coater can be controlled from room temperature to over 400°F. The temperature and speed are maintained so as to yield coated cloth suitably dried and with the desired degree of advancement. The average resin content is determined by weighing the coated cloth.

FABRICATION PROCEDURES

Fabrication procedures for each type of specimen are given in the following sections.

HOT-GAS FLOW SPECIMENS

Type A hot-gas flow specimens are machined from 3-1/2 x 1-7/8 x 1-1/2 inch laminated moldings, two specimens per molding. Types B and D specimens are machined from laminates. Type E specimens are machined from 3-1/2 x 1-7/8 x 1-1/2 inch moldings of 3/8-inch squares of impregnated cloth, two specimens per molding.

The prepregs for molding Type A hot-gas flow specimens are prepared by spatula or dip coating. After "B" staging, the material is cut into approximately 1-1/2 x 1-7/8 inch pieces using a blanking die. Groups of about 15 plies are stacked on pieces of aluminum foil and warmed for 20 minutes in a 160°F oven. A preforming tool is heated to 170°F in a press. All plies are placed in the preforming mold and pressure applied using a C-clamp. The preform is allowed to cool under pressure.

Preformed material was molded in the hot-gas flow mold using the molding conditions listed in Table 10. After postcure, the blank was cut in half using a diamond bandsaw and machined to final dimensions.

Type B and D specimens are machined from laminates at least 5/8-inch thick. Each laminate is sufficiently large to allow the machining of the required number of specimens. The laminates were molded using "B" staged prepreg and the molding conditions listed in Table 10. Specimens were rough cut prior to postcure to minimize the possibility of "blow-up". Diamond tools were used to machine the postcured pieces to final dimension.

Fabrication procedures for Type E specimens are fully described in Specific Specimen Procedures.

LAMINATES

When molding laminates, the "B" staged prepreg is cut into the proper size squares, randomized, stacked and wrapped in cellophane. The resulting layup is placed between 1/8-inch aluminum or 1/16-inch stainless steel cauls and loaded into the press. Laminates were cured using the molding conditions listed in Table 11. After postcure, the final resin content is determined. The laminate is then trimmed and squared by sawing with a diamond bandsaw. Finally, the density is calculated from the dimensions and weight of the specimen.

LAMINATED SQUARES

Fabrication procedures for laminated squares are the same as those for hot-gas flow specimens, types B and D. Laminates were molded and postcured using the conditions listed in Table 12.

PELLET SPECIMENS

When sufficient material is available, pellet specimens are machined from cylindrical moldings either 2 inches or 3-1/2 inches in diameter. The parts are usually molded at least 5/8-inch thick to ensure sufficient material for machining. Individual pellets are molded when not enough material is available for molding the larger discs. Pellet specimens machined from the larger discs would be expected to vary only slightly in composition compared with specimens individually molded. Before molding a large disc, a 3/4-inch diameter pellet is made to determine the molding characteristics of the prepreg. The charge weight for the large disc is then calculated using the following formula:

$$\text{Charge weight of large disc} = \frac{\left(\text{Diameter of large disc}\right)^2 \left(\text{Desired thickness of large disc}\right) \left(\text{Weight of 3/4-inch disc}\right)}{\left(\text{Diameter of 3/4-inch disc}\right)^2 \left(\text{Thickness of 3/4-inch disc}\right)}$$

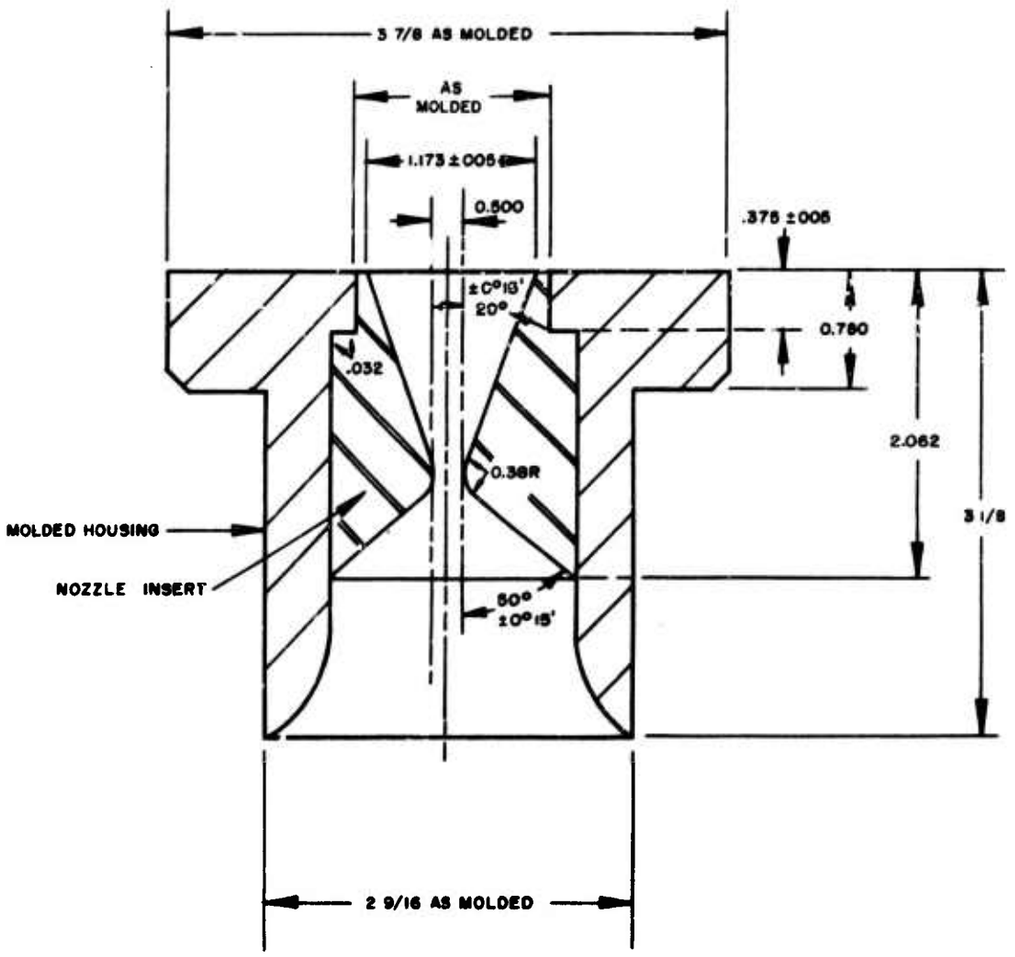
The number of plies needed to mold a laminated disc is calculated by first averaging the weights of five plies. The charge weight is then divided by the average weight per ply for the answer.

Discs were molded and postcured using the conditions listed in Table 13. The density is determined from the dimensions and weight. Pellet specimens are cut from large discs using a diamond bandsaw and all specimens are machined to final dimensions using a Carboloy cutter.

ROCKET NOZZLE SPECIMENS

The ASD No. 4 rocket nozzle assembly consists of a nozzle insert bonded into a molded phenolic housing as shown in Figure 2. All of the nozzle inserts fabricated during this report period were reinforced with fabric plies perpendicular to the nozzle axis and were machined from blank moldings or laminates with the exception of the Imidite 1850 nozzles (see Specific Specimen Preparation Procedures).

The method used to fabricate the nozzle insert blanks depended on the type of resin. Whenever possible, a high density, cylindrical blank was molded under high pressure in a compression mold (Figure 3). Some resin systems could not be cured while confined in a closed mold due to the release of excess volatiles which resulted in blistering and delamination. Materials containing such resins were laminated in an



NOZZLE INSERT OD MACHINE FOR SLIDE FIT INTO MOLDED HOUSING
INSERT BONDED INTO HOUSING WITH HAPEX 1208 (CATALYST 1213 14 PHR), CURE 1 HOUR AT ROOM TEMP
1 HOUR AT 200°F

Figure 2.

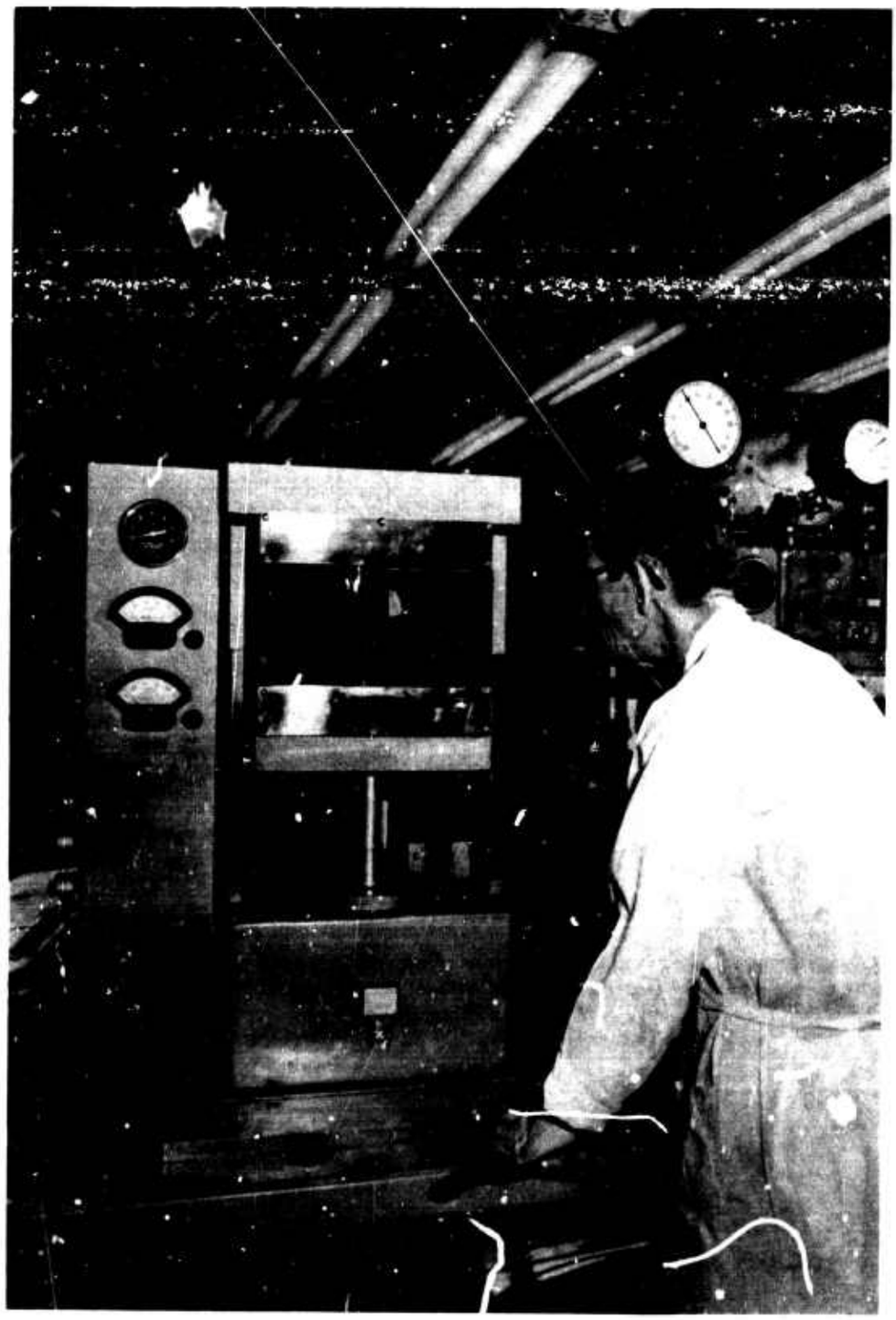


Figure 3. Molding of nozzle insert blank.

open laminating fixture which allowed the escape of excess volatiles during cure (Figure 4). Occasionally, it was necessary to fabricate a series of nozzle inserts and other specimens with virtually the same composition; the variation in resin content from molding to molding was onsidered unacceptably large. In this case, all of the required specimens, including the nozzle insert, were machined from a single, large, 1/2-inch thick laminate. The nozzle insert blank was made by bonding together several pieces to give a blank of the required thickness.

The nozzle housings were molded from MX2625, a heat-resistant, silica fiber and mineral-filled phenolic.

Nozzle inserts were molded and postcured using the conditions listed in Table 14. All of the internal dimensions of the inserts are machined. First, a 15/32-inch pilot hole is drilled using a tungsten carbide tool. The final configuration is then machined to the ASD No. 4 dimensions. A diamond tool on a tool post grinder is used for machining.

The density of the finished insert is determined by comparing its weight with that of an insert of known density molded from general purpose phenolic.

Nozzle inserts and nozzle housings are bonded together using Hapex 1208* containing 14-percent hardener. The bond is cured one hour at room temperature and one hour at 200°F.

SUPERSONIC PIPE SPECIMENS

Supersonic pipe specimens are cylinders 1.675 inches in diameter and 1.5 inches in height. These cylinders consist of plies of impregnated cloth either molded in the rocket nozzle mold or machined from blocks molded in the laminating fixture. Supersonic pipe specimens were molded and postcured using the conditions listed in Table 15. They were rough cut using a diamond bandsaw and when required, were machined to final dimensions using a Carboloy cutter.

*Hastings Plastics, 1704 Colorado Blvd., Santa Monica, California.

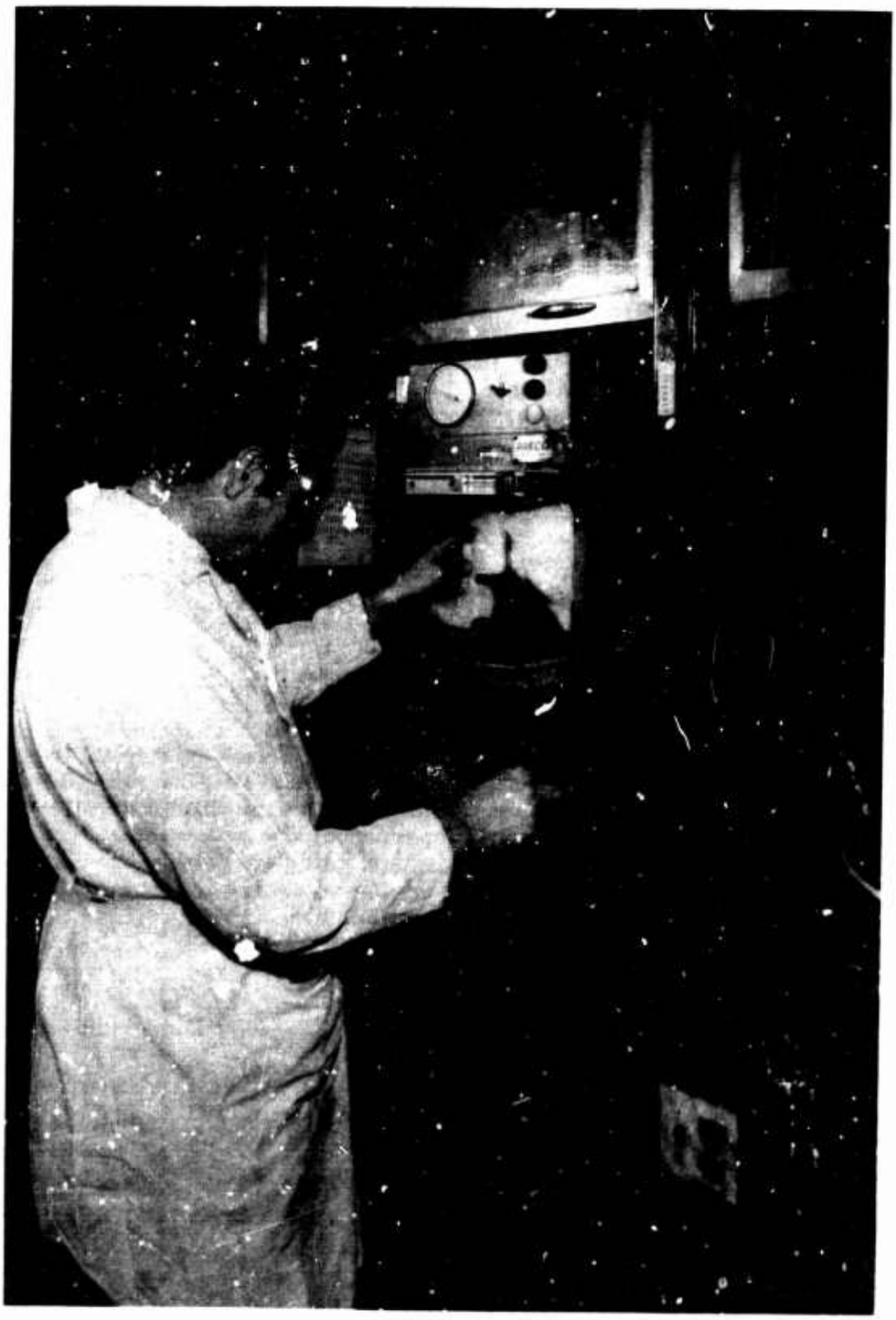


Figure 4. Lamination in open laminating fixture.

SPECIFIC SPECIMEN PREPARATION PROCEDURES

Certain specimens were fabricated by methods other than described in the previous section. Detailed procedure are listed below and are grouped according to similarity of preparation.

- Rocket Nozzle

Imidite 1850-181-112 E Glass Cloth

The molding material consisted of 181-112 E glass cloth coated with Imidite 1850 (resin content of 47.7 weight-percent) chopped into 1/2 inch squares. A charge weight of 140 grams was placed in the nozzle insert mold heated to a temperature of 600°F. The material was held at contact pressure for 30 seconds, after which time the pressure was increased slowly to 500 psi. After maintaining the pressure at 500 psi for 15 minutes, it was increased to 5000 psi. The temperature was increased at the same time to 700°F. Finally, the part was cured for three hours at 700°F, cooled to 200°F under pressure and removed from the mold.

The resin content of the nozzle insert, as molded, was 19.5 weight-percent. The part looked good in general. However, the thin area at the aft end was resin rich due apparently to the extreme fluidity of the resin during the initial application of pressure. This caused disproportionation of the resin and reinforcement to take place.

The nozzle insert was cut into two sections. One section was delivered in person to the Air Force Materials Laboratory Project Engineer.

The other section of the nozzle insert was post-cured as follows: 24 hours each at temperatures of 600°, 650°, 700° and 750°F, eight hours at 800°F and three hours at 700°F. During the postcure cycle, the insert was enclosed in an envelope made from thin sheet aluminum through which was passed a slow stream of dry nitrogen. From the appearance of the post-cured nozzle section, it was apparent that air was not effectively excluded during postcure. In some areas, the cured plastic matrix was burned away completely to the first layer of glass fibers. The molded section was not weighed prior to post-curing, so no estimate of the weight loss upon post-curing and the final resin content could be made.

- Rocket Nozzle

Data Sheet No. 173 — Imidite 1850 - Refrasil Cloth C100-48

Attempts were made in the preliminary molding of Imidite 1850 nozzles to mold inserts of uniform overall appearance with a resin content reasonably close to 35 weight-percent. The molding material was Imidite 1850 resin impregnated on 181-112 fiberglass in the form of 1/2-inch chopped squares. During molding, large quantities of volatile constituents were evolved and

considerable flow took place. The resin content as molded was approximately 20 weight-percent in all cases, although the contact time varied from 30 seconds to four minutes.

In the molding of the Imidite 1850-chopped Refrasil nozzle insert K-1-1, the final resin content after postcuring was 27.4 weight-percent. This somewhat higher value for the resin content can probably be attributed to the somewhat lower density of the Refrasil C100-48 (2.10 gm/cc) compared with that of the E glass cloth (2.57 gm/cc). With material which contains approximately 40 weight-percent resin solids, it is not possible to mold inserts with a desired resin content of 35 weight-percent. However, with the use of coated material containing considerably more resin solids along with some modification of the molding process, moldings with a higher resin content can be molded.

The Imidite 1850-Refrasil nozzle insert was postcured using the cycle listed in Table 14. The specimen was enclosed in an envelope made from thin sheet aluminum through which was passed a slow stream of dry nitrogen gas. Apparently, air was excluded fairly effectively during postcure since only moderate darkening took place. No evidence of resin burning away from the Refrasil cloth could be seen.

- Pellet Specimens
  - Data Sheet No. 235 — 91LD Resin-Polybenzimidazole Fibers
  - Data Sheet No. 236 — 91LD Resin-Polybenzimidazole Fibers

The specimens submitted using polybenzimidazole fibers and 91LD resin were prepared in the following manner. The first lot of yarn to be impregnated was oven dried at 240°F for two hours. Some yarn was then weighed on an analytical balance within 30 seconds after its removal from the desiccator. After obtaining the weight, the yarn was transferred to a piece of filter paper in a Buchner funnel. It was then impregnated with 91LD resin diluted with acetone and after a few minutes soak, the resin was removed by drawing a vacuum. After an oven dry at 160°F for 30 minutes, the yarn was "B" staged. The first three pellets were molded and then placed in the standard postcure (18 hours at 275°, 72 hours from 275° to 400°, 4 hours at 400°, and 7 hours cooling to 200°F). All three pellets were severely blistered upon removal.

Three more pellets were then molded and postcured over a longer period with a much more gradual rise time, (18 hours at 275°F, 170 hours from 275° to 400°F, four hours at 400°F and seven hours cooling to 200°F). These pellets had concave surfaces due to excessive shrinkage.

The remaining fibers themselves were then subjected to the second postcure cycle. Upon reweighing after cooling, they were found to have lost 10.4 percent of their original dry weight.

The postcured fibers were then vacuum impregnated, dried and "B" staged as described above and new pellet specimens molded. After molding the 45 weight-percent specimens, a 35 weight-percent pellet was molded. During the molding of the second 35 weight-percent pellet, it was found there was insufficient flow (probably due to advancing the resin too far during "B" staging). This resulted in a third part in the 45 weight-percent range. Before the four pellet specimens containing the postcured fibers could be postcured they were inadvertently shipped to Air Force Materials Laboratory.

Also included in this shipment for whatever value it might have was a molded pellet which contained only 26.4 weight-percent of 9ILD resin.

- Pellet Specimens
  - Data Sheet No. 331 — 91LD - Sapphire Wool Fibers
  - Data Sheet No. 333 — 91LD - Silicon Carbide Wool
  - Data Sheet No. 334 — 91LD-SC-70 Silicon Carbide Fiber
  - Data Sheet No. 335 — 91LD-T-70 Fiber Crystals

A weighed amount of fibers were soaked in a resin-acetone mixture under atmospheric conditions for at least one hour. After soaking, the container was placed under vacuum to remove the entrapped air which prevents complete coating of the fibers. After an hour under vacuum, the acetone completely boiled off. The coated fibers were air dried, oven dried and "B" staged. Sufficient material was not available for a two-inch diameter disc. Individual pellets were molded in the 3/4-inch diameter disc mold using conditions listed in Table 13. After postcure, the pellets were machined to required thickness.

- Pellet Specimens and Rocket Nozzle
  - Data Sheet No. 336 — 91LD - Rayon Fabric
  - Data Sheet No. 337 — 91LD - Rayon Fabric
  - Data Sheet No. 389 — 91LD - Rayon Silica Fabric

Rayon and rayon silica fabrics received from Air Force Materials Laboratory contained a finish which volatilized at postcure temperatures. An accurate resin content could not be determined until this substance was removed. Cloth was soaked in toluene, dried and weighed until a constant weight was obtained. Pellet specimens and a rocket nozzle were then fabricated using general procedures. (See also "Unsuccessful Experiments", Data Sheet Nos. 338 and 339.)

- Rocket Nozzles, Laminated Square Specimens and Pellet Specimens
  - Data Sheet Nos. 340, 343, 346 and 349 (Nozzles) — Imidite 4834

- Data Sheet Nos. 341, 344, 347 and 350 (Laminates) — Imidite 4824
- Data Sheet Nos. 342, 345, 348 and 351 (Pellets) — Imidite 4834

Narmco Materials Division preimpregnated the Imidite 1850 resin system on carbon cloth CCA-1 and designated the material Imidite 4834. Eight-ply laminates, four by six inches, were fabricated to determine the optimum molding conditions. Contact time is the major difference in molding Imidite-carbon cloth and Imidite-glass cloth laminates. Carbon cloth needs only 15 minutes while glass cloth requires 60.

Imidite 4834 was received from Narmco in two shipments, each with a different resin content. Plies from each shipment were alternated during layup of the laminate. The laminate was molded and postcured using conditions listed in Table 11.

Four nozzles were machined from blanks prepared by bonding several pieces of laminate together as follows:

Four pieces of 0.630 inch thick laminate, approximately 2 x 2 inches were prepared for bonding by sandblasting the faying surfaces. Two pieces were sandblasted on both sides, two on one side only. The four pieces were stacked together, the top and bottom surfaces of the stack being the surfaces which were not sandblasted. One piece of prepreg was inserted between each pair of faying surfaces. The stack was wrapped in several layers of aluminum foil to immobilize it and then bonded in a press using the following conditions:

- Temperature 700°F
- Pressure 1250 psi
- Cure time 180 minutes

The block was cooled to below 200°F before removing from the press. Eight laminated squares, 2 x 2 x 0.502 inches and eight pellets, 0.750 x 0.502 inches were machined from the remainder of the laminate.

Four groups of specimens consisting of two pellets, two laminates and one rocket nozzle were impregnated as requested with the following resins:

- Sylgard 182 (100 pbw Sylgard resin catalyzed with 10 pbw Sylgard 182 curing agent)
- R-7146 (100 pbw R-7146 resin catalyzed with 3 pbw dicumyl peroxide)
- DEN 438 (100 pbw DEN 438 resin catalyzed with 100 pbw of methyl nadic anhydride and 1 pbw of benzyldimethylamine

- QZ8-0903 (100 pbw QZ8-0903 catalyzed with 102 pbw methyl nadic anhydride and 1 pbw of benzyldimethylamine)

Specimens were impregnated twice, each cycle being performed as follows:

- Air was removed from the catalyzed resin under vacuum.
- Specimens were placed in a container and put under vacuum for four hours.
- Catalyzed resin was added to the container, under vacuum, until the specimens were immersed. The parts were soaked until all bubbling stopped and the resin became clear (approximately three hours).
- Specimens were removed from the vacuum and placed in a pressure pot for three hours at 850 psi.
- Specimens were removed from the pressure pot and excess resin removed by wiping with an acetone-soaked tissue.
- The impregnated specimens were cured at atmospheric pressure for four hours at 150°F, one hour at 212°F and one-half hour at 300°F.

- Pellet Specimens
  - Data Sheet No. 352 — Polyphenylene Sulfide - Refrasil cloth-carbon black

Polyphenylene sulfide pellets, reinforced with carbon cloth and containing 35 and 45 weight-percent resin, were molded successfully. However, the polyphenylene sulfide resin did not cure in combination with Refrasil. The Air Force project engineer suggested adding carbon black as a resin catalyst. A resin mixture was prepared containing 1 percent carbon black 452-00156 by grinding the powder into the resin with a mortar and pestle. Three 3/4-inch diameter pellets were successfully molded using dry powder layups heated to temperature by a red hot muffler heater around the cold mold. The reason is not known for the failure of the polyphenylene sulfide-Refrasil composite to cure without carbon black. Inhibition of the cure by the Refrasil or catalysis of the cure by the carbon are possible causes.

- Pellet Specimens and Rocket Nozzle
  - Data Sheet No. 373 — Chrome-P-Refrasil cloth C100-48
  - Data Sheet No. 374 — Chrome-P-Refrasil cloth C100-48
  - Data Sheet No. 375 — Chrome-P-carbon cloth CCA-1

Chrome-P resin composites were fabricated using general procedures. However, two molding temperatures were used. Initially, the composites were held at contact pressure for 15 minutes at 180°F. During this period considerable resin flow occurred. Following resin gelation, pressure was gradually applied. After two hours, the temperature was raised to 250°F and held for five hours.

The discs were postcured using the manufacturer's recommendations. The schedule consists of postcuring for one hour per inch of thickness at 150°, 200°, 250°, 300° and 350°F. Two hours at 400°F, per inch of thickness completes the postcure.

- Rocket Nozzles
  - Data Sheet No. 392 AFR-151 - carbon cloth CCA-1

Two AFR-151-carbon cloth nozzles were successfully molded using a method combining preimpregnated cloth with a dry powder layup. These materials were received 90 percent advanced from Narmco Materials Division which has now designated this resin as Imidite 2803. Extraction from the prepreg showed the cloth contained 44.2 weight-percent resin as received. However, a resin content of 46 weight-percent before molding was desired.

After blanking and randomizing the plies, 5 gm of powdered resin was equally distributed between the plies of each specimen being layed up. The layups were molded in a rocket nozzle mold at 600°F for 60 minutes followed by 120 minutes at 700°F. The standard Imidite postcure recommended by Narmco was used to postcure all the nozzle blanks. This postcure is not satisfactory for thick laminates since two molded blanks blew up during this cycle. A longer time at each temperature might eliminate this problem.

- Rocket Nozzles
  - Data Sheet No. 393 AFR-151-Refrasil Cloth Nozzles

Narmco Materials Division also prepared the AFR-151-Refrasil C100-3 cloth prepreg used to mold the nozzles. Resin burnoff of randomly selected samples showed an average resin content of 35.6 weight-percent in this prepreg. Powdered AFR-151 resin 90 percent advanced, was uniformly added between plies using a dry powder layup technique. This extra resin was required to obtain 40 weight-percent resin content after molding and postcure. Two nozzles were molded in the rocket nozzle mold using this technique and contained the required resin.

- Hot Gas Flow Specimens
  - Data Sheet 395a 91LD - carbon cloth CCA-1

A new type of hot gas flow specimen was requested by Air Force Materials Laboratory. This type is molded from 3/8-inch squares of impregnated reinforcement and has been designated as Type E. Sufficient carbon cloth was dried and spatula coated with 91LD resin to provide three moldings. After the normal air dry, oven dry and "B" staging, the material was blanked into 3/8-inch squares. The resulting molding material was thoroughly mixed to randomize the squares and then weighed into three approximately equal charges. Each charge was preformed in one operation to prevent fracture planes. These preforms were molded using the same conditions previously used to mold 91LD - carbon cloth laminate hot gas flow specimens.

After postcure, the blanks were cut in half prior to machining. Each piece had one or more cracks running most of its width. Further, X-ray analysis revealed the depth of these cracks extended almost to the surface of the parts.

These cracks could be caused by the following:

- Air entrapped during the preforming operation
- Volatiles unable to escape because of surface case hardening during the initial stages of molding
- Excessively long "B" staging time resulting in insufficient material flow

Two pieces broke during the machining operation but the other four were machined to dimension. These were shipped for whatever value they may have.

- Data Sheet 395b 91LD-carbon cloth CCA-1

Type E hot gas flow specimens were successfully prepared as follows. Pieces of carbon cloth were dried and spatula coated with 91LD resin. After air drying, oven drying and "B" staging, the material was blanked into 3/8-inch squares. The prepreg was thoroughly mixed to randomize the squares and then weighed out into charges for preforming.

Material was preformed under high pressure to minimize the amount of trapped air. Additionally the preforms were vacuum dried overnight to further remove volatiles. During molding, a step cure was used. The blanks were initially cured for 45-60 minutes at 200°F then 60 minutes at 300°F. The lower temperature prevented surface case hardening during the initial stages of molding allowing volatiles to escape.

X-ray analysis of the molded blanks revealed no cracks and after the standard postcure, they were machined into hot gas flow specimens.

- Pellet Specimens
  - Data Sheet No. 396 DEN 438 - boron fibers

The Air Force Materials Laboratory Project Engineer requested the fabrication of three pellet specimens of DEN 438 resin and boron fibers. These pellets were to contain the same volume percent resin as 35 weight-percent DEN 438, 65 weight-percent carbon cloth specimens.

Hughes received from Air Force Materials Laboratory several feet of inch-wide strips of boron fibers coated with DEN 438. These strips consisted of about 187 parallel fibers, one fiber thick. Resin contents were not supplied.

One-inch squares of fibers were blanked out and weighed. Soxhlet extractions were run to remove the "B" staged DEN 438 resin and to determine resin content. Additionally, specific gravity determinations

were to be run on extracted fibers to calculate average fiber density. However prolonged extraction with acetone, pyridine, piperidine and N, N-dimethyl formamide did not completely remove resin from fibers. Therefore accurate specific gravity and density values could not be determined.

The density for boron fibers used was selected from Technical Report AFML-TR-65-21, Boron Filaments and Composites, March 1965 (Confidential). A five mil average diameter fiber had a 2.53 gms/cc average density. Use of this value is reasonable since 100X magnification photographs of molded composites showed an average fiber diameter close to five mils.

The volume fraction of a 35 weight-percent DEN 438, 65 weight-percent carbon cloth CCA-1 composite was first calculated. From this, the resin content of the DEN 438-boron fiber composite was determined.

$$\text{Density} = \frac{\text{Weight}}{\text{Volume}} \quad \text{or} \quad \text{Volume} = \frac{\text{Weight}}{\text{Density}}$$

For DEN 438 resin, D = 1.25 gms/cc

$$\therefore V_{Resin} = \frac{35}{1.25} = 28.00$$

For Carbon cloth CCA-1, D = 1.84 gms/cc

$$\therefore V_{Cloth} = \frac{65}{1.84} = 35.33$$

$$V_{Total} = V_R + V_C = 63.33$$

Finally, for volume fraction of resin,

$$V_{Fraction} = \frac{28.00}{63.33} = 0.442$$

Since the DEN 438-boron fiber composite must have the same volume fractions of resin and reinforcement as the above, then

$$W_{Resin} = V_{Fraction\ of\ Resin} \times D_{Resin}$$

$$= 0.442 \times 1.25 = 0.552$$

$$W_{BF} = V_{Fraction\ of\ BF} \times D_{BF}$$

$$= 0.558 \times 2.53 = 1.412$$

$$W_{Total} = W_{Resin} + W_{BF} = 1.964$$

$$\text{Weight-Percent of Resin} = \frac{0.552}{1.964} \times 100 = 28.1$$

The one-inch wide strips of impregnated fibers were blanked into one-inch squares using a steel rule die. A layup was made by placing layers of fibers in a cold 1 x 1 inch mold. Each layer was rotated 90 degrees to the one below. No additional resin was added to the "B" staged fibers since incomplete resin extraction showed at least an average of 33.7 percent resin content. The parts were molded at 300°F for 120 minutes at 200 psi pressure. Sufficient material was available for three blocks.

Specimens are normally placed in a 275°F oven when beginning the standard postcure ("B" Schedule). An hour after the first block had been placed in this postcure, an examination revealed the specimen had cracked in several places. A possible explanation might be that the greater thermal conductivity of the boron fibers caused the part to crack when rapidly heated.

The block was impregnated with DEN 438 under 500 psi pressure and cured in a press at 300°F for 120 minutes at 200 psi pressure. This block was postcured with the other two. The oven was programmed from room temperature to 275°F over 16 hours, followed by the standard ("B" Schedule) postcure. The method was successful. Cracks were not evident after postcure, in any specimen.

Pellets were machined from the three blocks by first cutting off the corners using a diamond bandsaw. The diameter of two specimens was turned using diamond tools and the parts faced off using a Dumore grinder with a diamond wheel. The third pellet was completed by wet grinding to see if a better finish resulted. Comparison of dry and wet grinding techniques indicated dry grinding to be slightly better.

Resin content of the pellets had to be calculated differently since Soxhlet extraction of plies of impregnated fibers using various solvents proved unsuccessful. The approximate weight of boron fibers in each pellet was calculated using three assumptions:

1. The diameter of the fibers was five mils
2. There were 187 fibers per strip*
3. The density of the boron filaments was 2.53 gms/cc

The volume of a single 1-inch long five mil diameter fiber is:

*Determined by count of random plies.

$V_{Fiber} = \pi r^2 hc$ (c = conversion factor from cu. in. to cc.)

$= 3.1416 \times 0.0025 \times 0.0025 \times 1 \times 16.39$

$= 0.0003218$ cc

For the volume of a ply then:

$V_{Ply} = V_{Fiber} \times n$ (n = number of fibers in ply)

$= 0.0003218 \times 187$

$= 0.06018$ cc

The weight of a ply is found by:

$W_{Ply} = D_{Boron\ Fibers} \times V_{Ply}$

$= 2.53 \times 0.06018$

$= 0.1522$ gms

The weight of reinforcement is:

$W_{Reinforcement} = W_{Ply} \times N$ (N = number of plies in layup)

For Specimen

#1, N = 110; $W_{Reinforcement} = 0.1522 \times 110 = 16.74$ gms.

#2, N = 97; $W_{Reinforcement} = 0.1522 \times 97 = 14.76$ gms.

#3, N = 98; $W_{Reinforcement} = 0.1522 \times 98 = 14.92$ gms.

An approximate resin content for each specimen was then calculated in the usual manner. These values were in turn used to calculate the volume percent voids in resin.

- Rocket Nozzle
  - Data Sheet 403a — Skygard 700-Carbon Cloth Nozzle

Skygard 700 prepreg cannot be molded in the rocket nozzle mold because excessive volatiles are trapped in a closed mold, resulting in a poor molding. These volatiles escape when laminating the prepreg without a mold. A laminate, however, must be molded in a laminating fixture because of the small size of the plies and the thickness necessary to machine nozzles. This fixture keeps plies from slipping and spreading during the initial stages of molding while allowing escape of gases.

A 4 x 2-1/2 x 2-1/2 inch Skygard 700 - carbon cloth CCA-1 block was laminated using this technique. The prepreg contained 58.9 weight-percent resin before molding to compensate for the large resin flow and volatile loss during molding. Additionally, the plies were vacuum dried for 16 hours after "B" staging to remove the remaining N-methyl 2-pyrrolidone (NMP). A small amount of this high boiling point solvent can cause "blow-up" in a thick laminate. After molding, the block was cut in half prior to postcure to minimize the possibility of "blow-up". After postcure, one of the pieces broke in several places during machining. The other piece was successfully machined into a nozzle insert, bonded into a housing and shipped.

- Rocket Nozzles
  - Data Sheet No. 410 PH990-carbon cloth CCA-1

PH990 is a phosphonitrilic-modified phenolic powdered resin manufactured by El Monte Chemical Company. Company literature recommends the following formulation for optimum cured properties.

PH990	100 pbw
Hexamethylenetetramine	10 pbw
Magnesium oxide	10 pbw

Additionally, a cure temperature of 450°F was recommended during molding.

PH990 resin, hexamethylenetetramine and magnesium oxide were dry blended per vendor's instructions after which acetone was added for ease of coating. Carbon cloth, previously dried was spatula coated, air dried, oven dried and "B" staged using conventional procedures. Two blanks were molded which while solid, exhibited little flow. Additionally, the blanks weighed the same after postcure as before. The parts were then machined to the ASD No. 4 configuration and bonded into nozzle housings.

- Pellet Specimen
  - Data Sheet No. 413 DEN 438 - Boron Nitride Fibers

A small amount of boron nitride fibers in yarn form was received from the Air Force Materials Laboratory Project Engineer. Sufficient fibers were available to mold one 3/4-inch diameter by 1/2-inch pellet. The yarn was impregnated with DEN 438 resin by using the "soaking" technique. The required amount of resin was thinned with acetone and poured over the clustered yarn. Solvent was then removed by vacuum drying for three hours and oven drying for one hour at 160°F. After a ten minute "B" stage at 225°F, the impregnated yarn was molded in the 3/4-inch diameter pellet mold. The resulting pellet was postcured and machined to a 0.502 ±0.002 inch thickness.

The machined surfaces of the pellet showed that the yarn had not been uniformly impregnated. More uniform moldings can be obtained by chopping the fibers into short lengths prior to coating.

- Supersonic Pipe Specimens
  - Data Sheet No. 414 AFR 151-carbon cloth
  - Data Sheet No. 420 Skygard 700-graphite cloth
  - Data Sheet No. 425 91LD-carbon cloth

A Skygard 700-graphite cloth layup was molded in the rocket nozzle mold for use as a supersonic pipe specimen. This method however proved unsuccessful since the part fractured during removal. Excessive volatiles trapped by the closed mold caused the part to "blow up".

A 5 x 3 x 2 inch long Skygard 700-graphite cloth G1550 laminate was successfully prepared in the laminating fixture using the following technique. A stack of 5 x 3 inch plies was inserted between the rods of the bottom plate. The hat section punch preheated to 600°F, was slipped down over the rods until it rested on the stack. The fixture was placed in a 600°F press and pressure slowly applied as the material heated. Glass wool insulated the fixture and prepreg to help retain heat and after resin gelation, full pressure was applied. The laminate was cured for four hours and then gradually cooled to room temperature. Prior to postcure, the laminate was cut in half to minimize the possibility of "blow up". After postcure a supersonic pipe specimen was machined from each half.

The laminating fixture was also used to mold an AFR 151-graphite cloth laminate for supersonic pipe specimens. This laminate was unsatisfactory because of having to use a dry powder layup with 90 percent advanced resin. The Air Force Materials Laboratory Project Engineer therefore requested that the required specimens be fabricated from AFR 151-carbon cloth prepreg. A laminate was then successfully molded using the new fixture. Powdered AFR 151 resin was not needed since the resin content of the prepreg was sufficient. The resin in this prepreg was only 50 percent advanced and satisfactory flow was therefore obtained.

The Air Force Materials Laboratory Project Engineer further requested supersonic pipe specimens consisting of 91LD resin and carbon cloth CCA-1. These parts were fabricated from a laminate prepared in the laminating fixture.

- Supersonic Pipe Specimens
  - Data Sheet No. 418 91LD-graphite cloth
  - Data Sheet No. 419 91LD-graphite cloth

Sixteen supersonic pipe specimens were machined from a 9 x 8-1/2 x 2-1/4 inch laminate molded from 91LD resin and graphite cloth G1550. The cloth was impregnated with resin and "B" staged using the laboratory coater. For the first of two runs, the

resin pickup was 38.3 weight-percent, the cloth speed being 12 inches per minute. This speed was changed to 16 inches per minute for the second run to allow a greater percent resin pickup. The run therefore averaged 44.0 weight-percent resin content.

Each roll of cloth was cut into 9 x 8-1/2 inch plies which were then randomized. Ninety plies from each were intermixed, randomized, stacked and molded in a press without a laminating fixture. Such a fixture was unnecessary because the large size of the plies tended to prevent them from slipping in the press. The resin content of the stack prior to molding was 41.2 weight-percent. After molding, the laminate was cut into eight pieces to minimize the possibility of "blowing" during postcure. Following postcure, two specimens were machined from each segment.

Additionally two supersonic pipe specimens were prepared in the rocket nozzle mold. Discs were blanked out from the randomized plies of each of the two coating runs. The discs were intermixed, randomized, divided into equal parts and molded.

A comparison of the resin contents and densities of the molded specimens with those of the machined specimens show little difference. The slightly lower density of the laminated specimens results from the slightly higher resin content (see Table 15).

UNSUCCESSFUL EXPERIMENTS

- Pellet Specimens and Rocket Nozzle
  - Data Sheet No. 338 — 91LD-rayon silica fabric
  - Data Sheet No. 339 — 91LD-rayon silica fabric

Rayon silica fabrics received from Air Force Materials Laboratory contained a finish which volatilized at postcure temperatures. To remove this finish, the cloth was soaked in toluene, dried and weighed until a constant weight was obtained. Specimens were then fabricated using general procedures.

During the postcure of the rayon-silica fabric composites, a balancing motor burned out on the temperature controller regulating the oven. A two-inch diameter disc and a nozzle blank were subjected to 500°F for an unknown length of time. The disc lost 17.0 percent of its molded weight. Some loss was due to resin volatiles but the greater loss was from pyrolysis of the rayon silica fabric. An accurate resin content cannot be determined.

Three 3/4-inch diameter pellets were machined from the disc and shipped for what value they might have. Another 91LD-rayon silica fabric composite was prepared.

The nozzle blank lost 16.9 percent of its molded weight during postcure. The resulting pyrolysis of rayon silica fabric and resin weakened the blank. After the initial machining operations, the insert was bonded into a housing, to enable machining to be completed.

The rocket nozzle assembly was shipped for what value it might have.

- Pellet Specimens
  - Data Sheet No. 387 — PNP II - asbestos
  - Data Sheet No. 388 — PNP II - Refrasil C100-48

Representatives from Olin-Mathieson and Raybestos-Manhattan visited Hughes to help mold PNP II (carborane) composites. These composites consisted of PNP II - asbestos and PNP II - Refrasil cloth nozzle inserts. Material for the former was prepared by Raybestos-Manhattan and is designated as RM 9001. They reported the impregnated asbestos had a resin content of about 40 percent. The PNP II - Refrasil cloth composite was prepared by Hughes using a dry powder layup containing 55 percent resin before molding. The powdered resin was supplied by Olin-Mathieson.

PNP II - asbestos is a fluffy, fibrous material with a very high bulk factor. Due to this high bulk factor, it was necessary to preform the material at room temperature in the mold in which the material was to be cured. It was necessary to use a multiple loading technique in order to accommodate the entire mold charge. After each loading, the top surface of the preform was scratched in what later proved to be an unsuccessful attempt to prevent the formation of fracture planes during molding.

The mold containing the preformed material was placed between press platens previously heated to 700°F and a pressure of 6000 psi was applied to the material. The outside temperature of the mold was measured by means of an externally applied thermocouple. The mold was covered with glass wool to minimize heat losses.

About an hour after starting, the first traces of phosphine gas appeared, bursting into flame spontaneously upon contacting air. The material was estimated to be between 500° - 600°F for this phenomenon to take place. Shortly after, the heating element in the top platen failed. A muffler heater was connected to a variac and placed around the mold to prevent the temperature from dropping. The variac controlled the amount of heat supplied by the heater.

As the resin flowed and finally gelled a constant 6000 psi pressure was maintained. However, after gelation the heaters in the bottom platen failed and a second muffler heater-variac combination was used to keep up the temperature. This temperature could only be estimated from the outside since the mold had not been equipped to allow measurement of internal temperature. The inside temperature probably did not exceed 700 F. The part was cured for four hours and allowed to cool overnight.

The blank broke along a fracture plane during removal from the mold. Unfortunately, neither piece was large enough to be machined into a nozzle. However, the pieces were large enough to be machined into 3/4-inch diameter pellet specimens.

The PNP II - Refrasil C100-48 nozzle was prepared using a dry powder layup with a resin content of 55 percent before molding. Pressure was maintained on the part at 6000 psi while heating the mold to 700°F. Evolution of phosphine occurred about 1-1/2 hours after starting. Ten minutes later, the resin started to flow and a rapid drop in pressure occurred. Pressure must not be relieved at this critical stage or the evolution of gas gets out of hand. Therefore pressure was reapplied causing the resin to continue to flow. This flow under pressure continued until the punch landed on the mold. Further flow then resulted in a complete loss of pressure on the part. A few minutes later, the resin gelled and the cure continued for 60 minutes at 700°F followed by 60 minutes at 800°F. The mold was then cooled to room temperature overnight. The following morning the blank broke into three pieces while being removed.

Per instructions of Olin-Mathieson, the surface skin was removed from the pieces of both composites prior to postcure. These pieces were then placed in an oven and postcured in air using the following schedule recommended by Olin-Mathieson.

- 16 hours from room temperature to 250°F
- 12 hours at 250°F
- 250° to 800°F at a rate of 50°F every 12 hours
- 24 hours at 800°F
- Cooling to room temperature before removal from oven

The PNP II - asbestos appeared to be a tough solid composite after postcure. Using the prepreg resin content value supplied by Raybestos-Manhattan, the final resin content was 36.4 percent. The resin content of the PNP II - Refrasil composite could not be calculated. Sufficient small pieces had been lost when the blank was removed from the mold to make the original reinforcement weight inaccurate. Additionally the spongy nature of the composite made a density measurement meaningless.

Three 3/4-inch diameter pellets were machined from PNP II - asbestos. Two other pellets were machined from PNP II - Refrasil C100-48 cloth. The latter were forwarded to Air Force Materials Laboratory only for informational purposes.

- Pellets

Polyhydroxy Phenyl

Polyhydroxy phenyl resin specimens cannot be molded with reinforcements. At 850°-900°F the resin alone fuses into a hard tough molding. In combination with carbon cloth, the resin did not appear to wet the cloth or bond. Molding resulted in what appeared to be individual plies of cloth and powdered resin. The Air Force Materials Laboratory Project Engineer then requested the resin be used with various types of Refrasil cloth. Parts were attempted with C100-48 cloth, C100-28 cloth and "Irish Refrasil" cloth. The first two attempts yielded a mixture of plies of "black fabric" interspersed with a white powder. The last attempt resulted in a mixture of "black fabric" and green powder. At this point, the explanation was obvious. The "black fabric" was fused polyhydroxy resin which contained the imprint of the cloth. This resin did not wet or penetrate any type of cloth used. When fused, the resin ground the cloth plies to powder under the pressure applied. In the carbon cloth specimen, the black powdered cloth had been mistaken for the resin because the fused resin contained the imprint of this cloth.

MISCELLANEOUS NOTES

- Rocket Nozzle
  - Data Sheet No. 386 DEN 438-Refrasil cloth C100-48

A nozzle was molded of DEN 438 and Refrasil cloth C100-48 and contained 42 weight-percent of resin before postcure. After postcure, large cracks were present between plies. Examination of X-rays revealed several cracks penetrated almost completely through the molding.

A second blank was molded but the resin content of the prepreg was held to 39.1 weight-percent. Additionally, the prepreg was "B" staged for 60 minutes to keep resin flow to a minimum. Cracks were not present after postcure of this second part. Lower resin content and more advanced material resulted in a satisfactory nozzle.

- Pellet Specimens
  - Data Sheet No. 389 91LD-rayon silica fabric

These specimens are replacements for the pellet specimens shipped on Data Sheet No. 338. The other specimens were subjected to 500°F for an unknown length of time after the balancing motor of a temperature controller failed.

- Pellet Specimen
  - Data Sheet No. 390 91LD-fused silica fabric, coated with pyrolytic carbon

Sufficient material was available for only two pellets. These were molded individually in the 3/4-inch diameter pellet mold.

- Rocket Nozzles
  - Data Sheet No. 402 DEN 438 - carbon cloth

No problems were encountered in molding DEN 438-carbon cloth nozzle blanks containing 30 weight-percent resin. However these blanks "blew up" when subjected to the standard postcure*. Postcuring to 400°F using an extended schedule (100 hours from 275° to 400°F) did not help since specimens still cracked. DEN 438 is not a high temperature resin. After prolonged periods at elevated temperature, escaping volatiles might easily fracture the weakened resin. A considerably shorter postcure was run on one cylindrical blank to determine if this would eliminate the cracking. The part did not blow up. Two additional blanks were then successfully postcured using the same schedule.

*18 hours at 275°F, 72 hours from 275° to 400°F, four hours at 400°F, seven hours cooling to below 200°F.

This postcure cycle is as follows:

275°F	17 hours
275°-400°F	6 hours
400°F	1 hour
400°-200°F	7 hours

The cylindrical blanks were machined to ASD No. 4 configuration. However, the 0.500 inch throat dimension of the No. 2 specimen was inadvertently machined to 0.515 inch, 0.005 inch, over the allowable tolerance. This nozzle insert and the No. 1 insert were bonded into housings and shipped. A replacement was made for the No. 2 specimen.

A third rocket nozzle blank was 1.3 weight-percent over the allowable resin content. The Air Force Materials Laboratory Project Engineer requested this part be shipped without machining, as an extra specimen.

- Rocket Nozzles
  - Data Sheet No. 406 — Polyphenylene-carbon cloth CCA-1

Resin batch B2353-58 was used to spatula coat the carbon cloth. This batch was a blend of six batches and was an attempt to duplicate batch B2353-42 as requested.

- Laminates
  - Data Sheet No. 437 — Phenylphenol phenol formaldehyde-graphite cloth laminate
  - Data Sheet No. 441 — 91LD-graphite cloth laminate

Laminates were made with phenylphenol phenol formaldehyde impregnated graphite cloth and 91LD impregnated graphite cloth using general procedures. However, these laminates were cut into smaller pieces to prevent "blow-up" during postcure. The total weight of cut pieces was measured for each laminate and new reinforcement weights calculated. These weights were determined by finding the percent reinforcement in the laminates before postcure and multiplying this percentage by the weights of the cut pieces.

MATERIAL STUDIES

Polyphenylene Postcure

A test laminate containing 25 weight-percent resin was prepared from polyphenylene and E glass. Twenty-five 1/2 x 1/2 inch specimens were cut from this laminate. These specimens, in groups of five, were postcured in helium at temperatures of 450°, 500°, 600°, 700° and 800°F.

The following tables give the dimensional changes and weight loss for the postcured specimens and the postcure cycles.

The percent weight loss at 500° and 600°F is almost identical. The loss at 700°F, however, is more than double the loss at 600°F. Additionally, the first signs of resin burnoff exposing the cloth are visible at 700°F. Based on these results, 600°F was selected as the maximum postcure temperature

A rocket nozzle insert blank was then postcured to 600°F, but the blank severely blistered and contained large cracks. The maximum postcure temperature was dropped to 550°F and blistering and delamination were eliminated.

TABLE 1. POSTCURE SCHEDULES

Maximum Temperature	Temperature Schedule
450°F (control)	18 hrs at 275°F, 72 hrs from 275° to 450°F, 6 hrs at 450°F, 7 hrs cooling to below 200°F
500°F	18 hrs at 275°F, 90 hrs from 275° to 500°F, 7 hrs cooling to below 200°F
600°F	18 hrs at 275°F, 130 hrs from 275°F to 600°F, 7 hrs cooling to below 200°F
700°F	18 hrs at 275°F, 170 hrs from 275°F to 700°F, 7 hrs cooling to below 200°F
800°F	18 hrs at 275°F, 210 hrs from 275°F to 800°F, 7 hrs cooling to below 200°F

TABLE 2. DIMENSIONAL CHANGES AND WEIGHT LOSS FOR POSTCURED SPECIMENS

Specimen Number[1]	Before Postcure			Postcure Temp °F	After Postcure			% Wt[2] Loss
	Length Inches	Width Inches	Thickness Inches		Length Inches	Width Inches	Thickness Inches	
1a	0.5022	0.5042	0.1288	450 (Control)	0.5022	0.5042	0.1288	0.98
1b	0.5021	0.5069	.1270		0.5021	0.5069	0.1270	
2a	0.5031	0.5049	0.1289	500	0.5031	0.5049	0.1289	2.35
2b	0.5041	0.5030	0.1299		0.5041	0.5030	0.1289	
3a	0.5051	0.5028	0.1279	600	0.5051	0.5028	0.1272	2.41
3b	0.5050	0.5039	0.1298		0.5050	0.5039	0.1291	
4a	0.5030	0.5051	0.1031	700	0.5030	0.5050	0.1027	5.60
4b	0.5031	0.5042	0.1029		0.5031	0.5041	0.1026	
5a	0.5038	0.4978	0.1248	800	0.5036	0.4977	0.1236	7.21
5b	0.5055	0.5002	0.1249		0.5054	0.5001	0.1237	

[1]Two specimens in each group were randomly selected.

[2]Based on total weight loss for five specimens in group.

APPENDIX

FORMULATIONS USED WITH MULTIPART RESIN SYSTEMS

Material	Parts by Weight
DEN 438	
DEN 438 Resin	100
Methyl Nadic Anhydride	100
Benzyl Dimethyl Amine	1
QZ8-0903	
QZ8-0903 Resin	100
Methyl Nadic Anhydride	102
Benzyl Dimethyl Amine	1
R-7146	
R-7146 Resin	100
Dicumyl Peroxide	3
Sylgard 182	
Sylgard 182 Resin	100
Sylgard 182 Curing Agent	10
PH990	
PH990 Resin	100
Hexamethylenetetramine	10
Magnesium Oxide	10

RESIN SYNTHESES

All resins were synthesized by the Chemical Synthesis Group of the Materials Technology Department under the direction of Dr. Norman Bilow.

2,2-bis(p-hydroxyphenyl) Propane Phenol Formaldehyde

Batch No. B2353-20

A solution of sodium hydroxide (80 g., 2 mole) in water (200 ml) was added slowly to a slurry of 2,2-bis(p-hydroxyphenyl) propane (456 g., 2 mole) in formalin (37 percent CH_2O, 716 g., 8.8 mole). The temperature was maintained at 55°C for 11 hours. After cooling and acidifying the solution to a pH of 3.5 the precipitated polymer was collected by filtration and thoroughly dried in a vacuum oven. The resin was dissolved in ethanol and sufficient phenol formaldehyde homopolymer was added to yield a lacquer containing 51.2 percent 2,2-bis (p-hydroxyphenyl) propane formaldehyde, 21.4 percent phenol formaldehyde and 27.4 percent alcohol.

Abchar 413

Batch No. B2353-51

100 gm of polyphenylene were slurried in cold tetrachloroethane (100 cc) and were then warmed slowly toward reflux (3 different batches were mixed, but all were soluble in chlorobenzene and insoluble in 20 percent benzene + 80 percent hexane). When it began to appear too viscous trichloroethylene (100 cc) was added while stirring constantly. After all polymer was in solution, reflux continued for 1-2 hours then 200 ml $CHCl_3$ were added. A solution of the curing agent was prepared from xylyleneglycol (35 gm), p-toluene sulfonic acid (10.5 gm) and chloroform (350 ml) by refluxing the mixture for 24 hours while removing water. The curing agent solution was added to the polyphenylene solution and the mixture was heated at reflux for 24 hours. The lacquer was excellent.

Polyphenylene (Abchar 412)

Batch No. B2353-58*

Polyphenylene (92.5 g) (chlorobenzene soluble type) was dissolved in tetrachlorethane (140 ml) trichloroethylene (74 ml) and 1,1,1-trichloroethane (40 ml). The solution was heated at reflux for 30 hours then a solution of curing agent was added. The latter was prepared from recrystallized xylyleneglycol (44.8 g), p-toluene sulfonic acid monohydrate (13.4 g) and chloroform (470cc) by refluxing them for 22 hours while removing water. The polyphenylene-xylyleneglycol mixture was then heated at 70°C for 20 hours while stirring constantly.

Six batches were prepared and blended. Slightly shorter reaction periods were used than when preparing B2353-42. A new supply of recrystallized xylylene glycol was found to be more reactive than that previously available.

Abchar 700

This polymer is the intractable polyphenylene isolated from run C1414-27 reported in AFML-TR-65-8.

Polyphenylene Used In Lacquers

Batch Nos. C1414-68-1 and 67-1

One (1) mole biphenyl and 1 mole of terphenyl were melted together at 110°C. A mixture of 4 mole of $CuCl_2$ and 2 mole of aluminum chloride was then added gradually. After several hours the crude reaction mixture was treated with concentrated hydrochloric acid and thereafter was washed with water. After drying, the crude product was thoroughly washed with a hot mixture of benzene (20 percent) and naphtha (80 percent). When this wash was completed the polymer was extracted with hot benzene, then with hot chlorobenzene. These benzene and chlorobenzene soluble fractions were recovered by vacuum drying. Four portions had the following melting points: 160-175°, 168-203°, 172-222°, and 173-196°C. These portions were thoroughly mixed and were used in preparing lacquers.

This polyphenylene has a somewhat lower molecular weight than that of the polyphenylene usually used in Abchar 412, 413, and 414. It was synthesized by the standard Hughes process but was prepared for Hughes by a subcontractor.

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*This batch is a special modification attempting to duplicate Batch No. B2353-42.

Polyphenylene (1000-1500 MW)

*(Abchar 412 Resin - Special Solvent)

Batch No. C-1414-67-1

Solution A: Combined 50 g p-xylylene glycol
16.7 g p-toluenesulfonic acid
500 ml chloroform
Refluxed with stirring for 20 hours. Water removed with trap. Weight, 793.3 g.

Solution B: Added 103.2 g polyphenylene to stirred 155 ml of 1, 1, 2, 2-tetrachloroethane in small portions. Refluxed 1 hour at 135-140°C. Added 82 ml trichloroethylene and 44 ml 1, 1, 1-trichloroethane. Refluxed 26 hours.

Added Solution A to Solution B and refluxed for 20 hours at 69 ± 1°C with stirring.

Polyphenylene Resin (1000-1500 MW)

(Abchar 413; Batch C-1414-68-1)

Solution A: Combined 70 g p-xylyleneglycol
23.3 g p-toluenesulfonic acid
700 ml chloroform
Refluxed with stirring for 20 hours. Water removed with trap. Weight 1113.1 g.

Solution B: Added 210 g polyphenylene to stirred 315 ml of 1, 1, 2, 2-tetrachloroethane in small portions. Refluxed for 1 hour at 135-140°C. Added 945 ml trichloroethylene. Refluxed for about 4 hours.

Added Solution A to Solution B and refluxed for 20 hours at 76-78°C with stirring during most of this period.

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*This resin is the same as Batch 2353-42.

p-Phenylphenol Phenol Formaldehyde Resin

*Batch No. (C-1414-69-1)

A slurry was prepared by adding 244 gm. formalin (37% formaldehyde solution)to 340 gm. p-phenylphenol, 80 gm. sodium hydroxide in 180 ml water and 200 ml methanol. When the slurry was heated, it became a homogeneous solution and an exothermic reaction took place. Heating was continued as required to maintain a temperature of 71 ± 2°C for 8 hours. During this time a precipitate formed twice, and each time 100 ml of water was added to dissolve the solid material and keep everything in solution. This occurred at 1 hour 20 minutes and 3 hours 45 minutes from the start of heating. When the period of heating was completed, the solution was neutralized with 6N hydrochloric acid to a pH of 5. The precipitated product was collected and dried, first in air for 4 days, then in a vacuum over Drierite for 1 day. Weight, 400.4 g.

The product was dissolved in 100 ml ethanol, 150 ml benzene, and 500 ml acetone. After the solution was warmed and filtered, it was blended with 1206 g. 91 LD containing 64 percent solids, and 100 ml benzene was added. Solvent was then removed by distillation from the stirred solution over a period of 1 hour, during which time the boiling point rose from 74°C to 101°C. The cooled product was the desired resin.

2,7-Dihydroxynaphthalene Phenol Formaldehyde

**Batch No. C2943-1 (Abchar 200)

A mixture of 2,7-dihydroxynaphthalene (360 g., 2.25 mole), phenol (635 g., 6.75 mole), formalin (1017 g., 37 percent CH_2O, 12.5 mole), and barium hydroxide octahydrate (42 g.) was heated at 70°C for 2-2-1/4 hours. The solution was then cooled, neutralized with dilute sulfuric acid (10 percent). Water was removed from the solution by heating under vacuum (max. temperature 70°C). Alcohol was then added and the product again dried under vacuum. A phenol formaldehyde prepolymer (180 g., 64 percent solids or 115 g. solids) was added to the resultant lacquer. The final product weighed 1560 g. and was 77 percent solids.

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*This batch of resin is similar to Batch B2353-35 used to prepare specimens listed on Data Sheet No. 290.

**This batch of Abchar 200 is similar to Batch B2353-36 used to prepare the specimens listed on Data Sheet No. 291.

TABLE 1

PROPERTIES OF HOT GAS FLOW SPECIMENS

Data Sheet Number	Type of Specimen[1]	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
					Resin	Reinforcement	Volume Percent Voids in Resin	
353	Type D	9-40-C	1.32	55	42.4	57.6	24.3	91 LD resin with carbon cloth CCA-1 as reinforcement
354	Type D	9-40-R	1.50	65	37.7	62.3	19.0	91 LD resin with Refrasil cloth C100-48 as reinforcement.
357-1	Type A	9-40-C	1.36	75	41.1	58.9	21.4	91 LD resin with carbon cloth CCA-1 as reinforcement.
357-2			1.37	78	41.5	58.5	19.8	
358-1	Type A	9-40-R	1.60	65	40.8	59.2	5.56	91 LD resin with Refrasil cloth C100-48 as
358-2			1.57	67	40.8	59.2	8.73	reinforcement.
383	Type A	9-40-C	1.40	80	41.5	58.5	16.7	91 LD resin with carbon cloth CCA-1 as reinforcement
395a-1	Type E	9-40-C	1.40	78	42.7	57.3	15.9	91 LD resin with 3/8" squares of carbon cloth CCA-1
395a-2			1.37	78	42.6	57.4	19.0	as reinforcement.
395b-1	Type E	9-40-C	1.44	80	40.8	59.2	13.5	91 LD resin with 3/8" squares of carbon cloth CCA-1 as reinforcement.
395b-2			1.35	80	42.5	57.5	21.4	
395b-3			1.34	80	42.2	57.8	22.2	
451/437	Type D	PPP-35-GU	1.36	35	36.2	63.8	26.9	Phenylphenol phenol formaldehyde resin with graphite cloth G1550 as reinforcement.
457/441	Type D	9-35-GU	1.32	50	36.4	63.6	34.3	LD resin with graphite cloth G1550 as reinforcement.

1 Dimensions of Hot Gas Flow Specimens are as follows:

Type A 3-1/3" x 1-3/4" x 1/2" (each ply 1-3/4" x 1/2")
Type D 3-1/3" x 2" x 1/2" (each ply 3-1/3" x 2")
Type E 3-1/3" x 1-3/4" x 1/2" (3/8" squares of reinforcement)

TABLE 2

PROPERTIES OF LAMINATES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
332[1]	N-35-C	1.24	46	30.8	69.2	46.2	Imidite 1850 resin syst with carbon cloth CCA-1 as reinforcement.
368	PP413-35-G	1.75	38	34.9	65.1	6.3	Polyphenylene (... har 413) resin with glass cloth, Style 181, A1100 finish as reinforcement.
437[2]	PPP-35-GU	-	-	36.2	63.8	-	Phenylphenol phenol formaldehyde resin with graphite cloth G1550 as reinforcement.
441[3]	9-35-GU	-	-	36.4	63.6	-	91LD resin with graphite cloth G1550 as reinforcement.

1 The Imidite 1850 resin system-carbon cloth CCA-1 laminate fabricated on Data Sheet No. 332 was machined into laminated squares and pellets and bonded and machined into rocket nozzles. These specimens were impregnated with one of 4 resins. The weight-percent of the impregnating resin appears in "Composition Wt-% Resin" column for Data Sheet Nos. 340, 341, 342, 343, 344, 345, 346, 347, 348, 349, 350 and 351.

2 The phenylphenol phenol formaldehyde-graphite cloth G1550 laminate fabricated on Data Sheet No. 437 was machined into hot gas flow specimens (Data Sheet No. 451), laminated squares (Data Sheet No. 452) and pellets (Data Sheet No. 453.)

3 The 91LD-graphite cloth G1550 laminate fabricated on Data Sheet No. 441 was machined into hot gas flow specimens (Data Sheet No. 457), laminated squares (Data Sheet No. 458) and pellets (Data Sheet No. 459).

TABLE 3

PROPERTIES OF LAMINATED SQUARES

Data Sheet Number	Code Number	Density gm/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
341/332	N-35-C(SY)		-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with Sylgard 182 resin.
L-SY-1				8.95[1]	-	-	
L-SY-2				9.39[1]			
344/332	N-35-C(S)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with R-7146 resin.
L-S-1				7.69[1]			
L-S-2				8.04[1]			
347/332	N-35-C(D)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with DEN 438 resin.
L-D-1		-	-	3.43[1]	-	-	
L-D-2				3.61[1]			
350/332	N-35-C(SE)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with QZ8-0903 resin.
L-SE-1		-	-	9.28[1]	-	-	
L-SE-2				8.62[1]			
452/437	PPP-35-GU	1.36	35	36.2	63.8	26.9	Phenylphenol phenol formaldehyde resin with graphite cloth G1550 as reinforcement.
458/441	9-35-GU	1.32	50	36.4	63.6	34.3	91 LD resin with graphite cloth G1550 as reinforcement.

1 Values listed are weight-percent of impregnated resin.

TABLE 1

PROPERTIES OF PELLETS SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight Percent			Description of Material
				Resin	Reinforcement	Volume Percent Void Resin	
235-1	9-45-PBIF	1.26	50	50.2	49.8	-	91 LD resin with polybenzimidazole fibers as reinforcement.
235-2		1.26	50	49.8	50.2	-	
235-3		1.27	50	49.4	50.6	-	
236	9-35-PBIF	1.27	50	36.3	63.7	-	91 LD resin with polybenzimidazole fibers as reinforcement.
330	9-35-GCB	1.35	30	37.0	63.0	20.6	91 LD resin with graphite cloth G1550 coated with 1μ pyrolytic graphite-boron alloy as reinforcement.
331-1	9-35-SW	2.08	82	35.5	64.5	-	91 LD resin with sapphire wool fibers (1-3μ diameter) as reinforcement.
331-2		2.18	80	35.1	64.9	-	
333-1	9-35-SCW	1.75	85	36.7	63.3	-	91 LD resin with silicon carbide wool fibers (2-8μ diameter) as reinforcement.
333-2		1.76	87	37.0	63.0	-	
334-1	9-35-SC70	2.13	80	37.0	63.0	-	91 LD resin with SC-70 silicon carbide fibers and bulk crystal mix (1-5μ diameter) as reinforcement.
334-2		2.03	80	37.2	62.8	-	
335-1	9-35-T70	1.92	82	37.8	62.2	-	91 LD resin with T-70 fiber crystals (aluminum nitride and aluminum oxide fibers) as reinforcement.
335-2		2.03	82	37.5	62.5	-	
336	9-35-RF	1.33	65	36.0	64.0	-	91 LD resin with rayon fabric as reinforcement.
338	9-35-RSF	1.27	40	26.2[1]	-	-	91 LD resin with rayon silica fabric as reinforcement.
342/332	N-35-C(SY)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with Sylgard 182 resin.
P-SY-1				8.41[2]	-	-	
P-SY-2				8.02[2]			
345/332	N-35-C(S)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with R-7146 resin.
P-S-1		-	-	8.24[2]	-	-	
P-S-2				7.20[2]			

TABLE 4 (CONTINUED)
PROPERTIES OF PELLETS SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
348/332	N-35-C(D)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with DEN 438 resin.
P-D-1		-	-	2.86_2	-	-	
P-D-2				3.46_2			
351/332	N-35-C(SE)	-	-		-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with QZ8-0903 resin.
P-SE-1		-	-	8.85_2	-	-	
P-SE-2				9.83_2			
352-1	PS-35-RCB	1.68	30	36.2	63.8	-	Polyphenylene sulfide resin containing 1% carbon black 452-00156 with Refrasil cloth C100-48 as reinforcement.
352-2		1.70	32	36.7	63.3		
352-3		1.71	35	37.0	63.0		
360	9-35-CLA	1.40	70	35.3	64.7	23.0	91 esin with carbon cloth CCA-1, low alkalinity, as reinforcement.
369	PP413-35-C	1.30	30	38.6	61.4	24.6	Por ylene (Abchar 413) resin with carbon cloth CCA-1 as reinforcement.
370	9PP-45-C	1.34	68	91LD 30.2 PP 15.9	53.9	-	91 LD containing polyphenylene resin (Abchar 700) with carbon cloth CCA-1 as reinforcement.
374	CP-35-R	1.61	73	34.4	65.6		Chr e-P resin with Refrasil C100-48 as reinforcement.
375	CP-35-C	1.30	65	33.8	66.2	-	Chrome-P resin with carbon cloth CCA-1 as reinforcement.
376	9-35-GCB (2.5)	1.42	30	35.7	64.3	26.2	91 LD resin with graphite cloth G1550, coated with 2.5μ pyrolytic graphite-boron alloy.
385	9-35-CSF	1.49	75	35.6	64.4	-	91 LD resin with carbon silica fabric as reinforcement.
387	PNP II-40-A	1.72	24	36.4	63.6	-	PNP II resin with asbestos as reinforcement.
388	PNP II-40-R	-	-	-	-	-	PNP II resin with Refrasil cloth C100-48 as reinforcement.
389	9-35-RSF	1.39	55	34.4	65.6	-	91 LD resin with rayon silica fabric as reinforcement.
390-1	9-35-FSF (PC)	1.62	66	35.2	64.8	-	91 LD resin with fused silica fabric, coated with pyrolytic carbon, as reinforcement.
390-2		1.64	66	35.3	64.7		

TABLE 4 (CONTINUED)
PROPERTIES OF PELLETS SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
396-1[4]	D-30-BF	1.93	90	27.6	72.4	4.8	DEN 438 resin with boron fibers as reinforcement.
396-2[4]		1.94	85	26.2	73.8	6.4	
396-3[4]		1.97	90	26.1	73.9	3.2	
413	D-35-BN	1.47	60	38.4	61.6	-	DEN 438 resin with boron nitride fibers as reinforcement.
422	9-35-QF	1.73	73	33.6	66.4	-	91 LD resin with quartz fabric 581 as reinforcement.
453/437	PPP-35-GU	1.35	35	36.2	63.8	27.9	Phenylphenol phenol formaldehyde resin with graphite cloth G1550 as reinforcement.
459/441	9-35-GU	1.54	50	36.4	63.6	32.5	91 LD resin with graphite cloth G1550 as reinforcement.

1 During postcure, the balancing motor on a temperature controller burned out. The disc was subjected to 500° F for an unknown length of time and lost 17.0% of its molded weight. While some loss was due to resin volatiles, the greater part was due to pyrolysis of the fabric. An accurate resin content cannot be determined.

2 Values listed are weight-percent of impregnated resin.

3 The parts were machined from a nozzle blank which fragmented on removal from the mold. Loss of small fragments made an accurate postcured resin content impossible to determine. Additionally, an accurate density and Barcol hardness could not be determined because of excessive porosity.

4 Resin and reinforcement contents and vol-% voids in resin values are approximate and are based on a density for 5 mil boron fibers of 2.53 gms/cc.

TABLE 5
PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
173 K-1	N-35-R	1.59	62	27.4	72.6	26.2	Imidite 1850 resin system with 1/2" x 1/2" squares of Refrasil cloth C100-48 as reinforcement
337 K-38	9-40-RF	1.34	68	41.3	58.7	-	91 LD resin with rayon fabric as reinforcement.
339 K-39	9-40-RSF	1.28	45	21.6_1	-	-	91 LD resin with rayon silica fabric as reinforcement.
340/332 K-40-1	N-35-C(SY)	-	-	8.14_2	-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with Sylgard 182 resin.
343/332 K-41-1	N-35-C(S)	-	-	7.07_2	-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with R-7146 resin.
346/332 K-42-1	N-35-C(D)	-	-	6.55_2	-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with DEN 438 resin.
349/332 K-43-1	N-35-C(SE)	-	-	5.84_2	-	-	Imidite 1850 resin system with carbon cloth CCA-1 as reinforcement and impregnated with QZ8-0903 resin.
356 K-45	N151-40-G	1.42	45	43.6	56.4	30.8	AFR-151(40) resin with glass cloth, style 181, A1100 finish as reinforcement.
359 K-46-1 K-46-2	9-40-CLA	 1.41 1.41	 70 65	 42.6 42.8	 57.4 57.2	 15.1 15.1	91 LD resin with carbon cloth CCA-1, low alkalinity, as reinforcement.
364 K-44	N151-40-R	1.64	65	40.5	59.5	4.6	AFR-151(38) resin with Refrasil cloth C100-48 as reinforcement.
372 K-51-1 K-51-2	PP(413)-40-C	 1.36 1.34	 55 55	 37.6 38.3	 62.4 61.7	 19.6 21.0	Polyphenylene resin (Abchar 413) with carbon cloth CCA-1 as reinforcement.

TABLE 5 (CONTINUED)
PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
373	CP-40-R					-	Chrome-P resin with Refrasil cloth C100-48 as reinforcement.
K-48		1.64	68	38.7	61.3		
384	9-40-CSF					-	91 LD resin with carbon silica fabric as reinforcement.
K-49-1				41.9	58.1	-	
K-49-2		1.53	78	40.2	59.8		
386	D-40-R						DEN 438 resin with Refrasil C100-48 as reinforcement.
K-50		1.60	70	38.9	61.1	6.6	
392	N151-40-C						AFR 151 resin with carbon cloth CCA-1 as reinforcement.
K-52-1		1.35	78	38.0	62.0	28.2	
K-52-2		1.29	80	41.0	59.0	31.3	
393	N151-40-R						AFR-151 resin with Refrasil cloth C100-48 as reinforcement.
K-53-1		1.60	50	40.4	59.6	9.9	
K-53-2		1.59	50	39.8	60.2	11.4	
399a	9-40-C						91 LD resin with carbon cloth CCA-1 as reinforcement.
K-47-1		1.40	80	42.0	58.0	16.7	
K-47-2		1.41	75	41.4	58.6	15.9	
399b	9-40-C						91 LD resin with carbon cloth CCA-1 as reinforcement.
K-47-3		1.42	79	43.6	56.4	12.7	
400	9-40-GU						91 LD resin with graphite cloth G1550 (uncoated) as reinforcement.
K-58-1		1.35	53	40.3	59.7	27.7	
K-58-2		1.35	45	39.7	60.3	28.2	
401	9-40-R	1.63	75	41.1	58.9	2.4	91 LD resin with Refrasil cloth C100-48 as reinforcement.
K-59-1		1.61	76	40.3	59.7	4.8	
K-59-2		1.62	75	38.4	61.6	6.3	
K-59-3							
402a	D-30-C						DEN 438 resin with carbon cloth CCA-1 as reinforcement.
K-60-1		1.36	75	30.3	69.7	31.8	
K-60-2		1.34	76	31.4	68.6	32.6	

TABLE 5 (CONTINUED)
PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
402b$_3$	D-30-C	1.39	79	33.3	66.7	25.2	DEN 438 resin with ca[illegible] cloth CCA-1 as reinforcement.
402c K-60-3	D-30-C	1.34	60	28.8	71.2	35.8	DEN 438 resin with carbon cloth CCA-1 as reinforcement.
403a K-61-1	SG7-40-C	1.25	51	41.2	58.8	-	Ek, ard 790 resin wi[illegible] carbon cloth CCA-1 as reinforcement.
406 K-64-1 K-64-2	PP(412)-40-C	 1.31 1.31	 40 40	 38.0 38.2	 62.0 61.8	 25.8 25.5	Polyphenylene resin (Abchar 412) with carbon cloth CCA-1 as reinforcement.
410 K-68-1 K-68-2	PH9-40-C	 1.33 1.39	 59 60	 40.4 39.9	 59.6 60.1	 - -	PH 990 resin with carbon cloth CCA-1 as reinforcement.
423 K-71-1 K-71-2	PPF-40-C	 1.34 1.36	 78 81	 40.5 40.8	 59.5 59.2	 - -	2,2-bis (p-hydroxyphenyl) propane-phenol formaldehyde resin with carbon cloth CCA-1 as reinforcement.
424a K-72-1	PPF-40-R	1.52	68	42.2	57.8	-	2,2-bis (p-hydroxyphenyl) propane-phenol formaldehyde resin with Refrasil cloth C100-48 as reinforcement.
424b K-72-2	PPF-40-R	1.59	65	39.4	60.6	-	2,2-bis (p-hydroxyphenyl) propane-phe[illegible]l formaldeh[illegible] resin with Refrasil cloth C100-48 as reinforcement.

1 During postcure, the balancing motor on a temperature controller burned out. The nozzle blank was subjected to 500° F for an unknown length of time and lost 16.9% of its molded weight. While some loss was due to resin volatiles, the greater part was due to pyrolysis of the fabric. An accurate resin content cannot be determined.

2 Values listed are weight-percent of impregnated resin.

3 This specimen was a rocket nozzle blank whose resin content was 1.3 wt-% over the allowable tolerance. The unmachined cylindrical blank was shipped as an extra specimen.

TABLE 6

PROPERTIES OF SUPERSONIC PIPE SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material
				Resin	Reinforcement	Volume Percent Voids in Resin	
414	AFR151-35-C	1.16	50	33.6	66.4	48.8	AFR-151 resin with carbon cloth CCA-1 as reinforcement.
415-1	PP(412)-35-GU	1.25	35	36.6	63.4	-	Polyphenylene resin (Abchar 412) with graphite cloth G1550 (uncoated) as reinforcement.
415-2		1.28	20	36.3	63.7	-	
416-1	PPP-35-GU	1.34	52	37.2	62.8	-	Phenylphenol phenol formaldehyde resin with graphite cloth G1550 (uncoated) as reinforcement.
416-2		1.34	55	37.4	62.6	-	
417-1	DN-35-GU	1.32	48	35.3	64.7	-	2, 7 dihydroxynaphthalene phenol formaldehyde resin with graphite cloth G1550 (uncoated) as reinforcement.
417-2		1.32	48	35.6	64.4	-	
418-1	9-35-GU	1.31	38	36.3	63.7	-	91 LD resin with graphite cloth G1550 (uncoated) as reinforcement.
418-2		1.34	38	35.5	64.5	-	
419	9-35-GU	1.26	38	37.3	62.7	-	91 LD resin with graphite cloth G1550 (uncoated) as reinforcement.
420	SG7-35-GU	1.21	35	32.4	67.6	-	Skygard 700 resin with graphite cloth G1550 (uncoated) as reinforcement.
421-1	9-35-GC	1.40	38	37.6	62.4	-	91 LD resin with graphite cloth G1550 coated with 1μ pyrolytic graphite as reinforcement.
421-2		1.42	36	36.7	63.3	-	
425	9-35-C	1.30	60	35.5	64.5	32.7	91 LD resin with carbon cloth CCA-1 as reinforcement.

TABLE 7

TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Code	RTD Letter Reference	Date of Shipment	Detailed Letter Report	
					Reference Number	Date
173	Nozzle	N-35-R	7 Nov 1962 (1c)	19 Sept 63	2748.1/361	10 Oct 1963
235	Pellet	9-45-PBIF	25 Oct 63 (7b)	7 Feb 64	2748.1/462	9 Mar 1964
236	Pellet	9-35-PBIF	25 Oct 63 (7a)	7 Feb 64	2748.1/462	9 Mar 1964
330	Pellet	9-35-GCB	19 Aug 64 Appendix B(1c)	20 Apr 65	2748.1/685	21 May 1965
331	Pellet	9-35-SW	18 Sept 64 (1a)	20 Apr 65	2748.1/685	21 May 1965
333	Pellet	9-35-SCW	18 Sept 64 (1b)	20 Apr 65	2748.1/685	21 May 1965
334	Pellet	9-35-SC70	18 Sept 64 (1c)	20 Apr 65	2748.1/685	21 May 1965
335	Pellet	9-35-T70	18 Sept 64 (1d)	20 Apr 65	2748.1/685	21 May 1965
336	Pellet	9-35-RF	19 Nov 64 (2a)	20 Apr 65	2748.1/685	21 May 1965
337	Nozzle	9-40-RF	19 Nov 64 (2c)	20 Apr 65	2748.1/685	21 May 1965
338	Pellet	9-35-RSF	19 Nov 64 (2b)	3 Jun 65	2748.1/700	14 Jun 1965
339	Nozzle	9-40-RSF	19 Nov 64 (2d)	21 Jun 65	2748.1/720	25 Jun 1965
340	Nozzle	N-35-C(SY)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
341	Lam. Square	N-35-C(SY)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
342	Pellet	N-35-C(SY)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
343	Nozzle	N-35-C(S)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
344	Lam. Square	N-35-C(S)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
345	Pellet	N-35-C(S)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
346	Nozzle	N-35-C(D)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
347	Lam. Square	N-35-C(D)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
348	Pellet	N-35-C(D)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
349	Nozzle	N-35-C(SE)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
350	Lam. Square	N-35-C(SE)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
351	Pellet	N-35-C(SE)	19 Mar 64 (2)	4 Mar 65	2748.1/685	21 May 1965
352	Pellet	PS-35-RCB	22 Jun 64 (5c) as amended by Minutes of Meeting 16-17 Feb 65(10)	20 Apr 65	2748.1/685	21 May 1965
353	Hot Gas Flow	9-40-C	Telecon from P. Pirrung 24 Feb 65, and 4 Mar 65, Appendix A(1c)	24 Mar 65	2748.1/685	21 May 1965
354	Hot Gas Flow	9-40-R	Telecon from P. Pirrung 24 Feb 65 and 4 Mar 65, Appendix A(1d)	24 Mar 65	2748.1/685	21 May 1965
356	Nozzle	N151-40-G	4 Mar 65 (1)	4 May 65	2748.1/685	21 May 1965
357	Hot Gas Flow	9-40-C	4 Mar 65 Appendix A(1a)	4 May 65	2748.1/685	21 May 1965
358	Hot Gas Flow	9-40-R	4 Mar 65 Appendix A(1b)	4 May 65	2748.1/685	21 May 1965
359	Nozzle	9-40-CLA	4 Mar 65 (4)	14 May 65	2748.1/685	21 May 1965
360	Pellet	9-35-CLA	4 Mar 65 Appendix C(1c)	14 May 65	2748.1/685	21 May 1965
364	Nozzle	N151-40-R	4 Mar 65 (1) and telecon from P. Pirrung 26 Mar 65	20 Apr 65	2748.1/685	21 May 1965
368	Laminate	PP413-35-G	19 Nov 64 (3b)	14 May 65	2748.1/685	21 May 1965
369	Pellet	PP413-35-C	19 Nov 64 (3c)	14 May 65	2748.1/685	21 May 1965

TABLE 7 (CONTINUED)
TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Code	RTD Letter Reference	Date of Shipment	Detailed Letter Report	
					Reference Number	Date
370	Pellet	9PP-45-C	19 Nov 64 (4)	14 May 65	2748.1/685	21 May 1965
372	Nozzle	PP(413)-40-C	19 Nov 64 (3a)	10 Jan 66	2748.1/860	14 Feb 1966
373	Nozzle	CP-40-R	19 Nov 64 (5a)	21 Jun 65	2748.1/720	25 Jun 1965
374	Pellet	CP-35-R	19 Nov 64 (5b)	3 Jun 65	2748.1/700	14 Jun 1965
375	Pellet	CP-35-C	19 Nov 64 (5c)	3 Jun 65	2748.1/700	14 Jun 1965
376	Pellet	9-35-GCB(2.5)	4 Mar 65 Appendix C(1a)	20 Jul 65	2748.1/745	4 Aug 1965
383	Hot Gas Flow	9-40-C	4 Mar 65 Appendix A(1a)	21 Jun 65	2748.1/720	25 Jun 1965
384	Nozzle	9-40-CSF	Telecon from P. Pirrung 5 May 65 and 7 July 65, App A(1b)	3 Jun 65 9 Jun 65	2748.1/700 2748.1/712	14 Jun 1965 16 Jun 1965
385	Pellet	9-35-CSF	Telecon from P. Pirrung 5 May 65 and 7 July 65, App A(1a)	3 Jun 65	2748.1/700	14 Jun 1965
386	Nozzle	D-40-R	Minutes of Meeting 17 May 65	9 Jun 65	2748.1/712	16 Jun 1965
387	Pellet	PNP II-40-A	13 May 65 (to Olin Mathieson)	27 Aug 65	2748.1/765	23 Sept 1965
388	Pellet	PNP II-40-R	13 May 65 (to Olin Mathieson)	27 Aug 65	2748.1/765	23 Sept 1965
389[1]	Pellet	9-35-RSF	29 Nov 64 (2b)	20 Jul 65	2748.1/745	4 Aug 1965
390	Pellet	9-35-FSF (PC)	7 Jul 65 (5)	13 Aug 65	2748.1/758	8 Sept 1965
392	Nozzle	N151-40-C	15 Apr 65 (3c)	1 Oct 65	2748.1/785	26 Oct 1965
393	Nozzle	N151-40-R	15 Apr 65 (3d)	26 Jan 66	2748.1/860	14 Feb 1966
395a	Hot Gas Flow	9-40-C	11 Aug 65, Appendix B(1a)	27 Aug 65	2748.1/765	23 Sept 1965
395b[2]	Hot Gas Flow	9-40-C	11 Aug 65, Appendix B(1a)	4 Oct 65	2748.1/785	26 Oct 1965
396	Pellet	D-30-BF	7 Jul 65, Appendix A(2a) and Min. of Meeting 17 May 65 (8)	13 Aug 65	2748.1/758	8 Sept 1965
399a	Nozzle	9-40-C	11 Aug 65, Appendix D(1c)	17 Sept 65	2748.1/765	23 Sept 1965
399b	Nozzle	9-40-C	11 Aug 65, Appendix D(1c)	1 Oct 65	2748.1/785	26 Oct 1965
400	Nozzle	9-40-GU	11 Aug 65, Appendix D(1d)	15 Oct 65	2748.1/785	26 Oct 1965
401	Nozzle	9-40-R	11 Aug 65, Appendix D(1e)	26 Jan 66	2748.1/860	14 Feb 1966
402a	Nozzle	D-30-C	11 Aug 65, Appendix D(1f)	7 Dec 65	2748.1/815	8 Dec 1965
402b	Cylindrical Blank	D-30-C	11 Aug 65, Appendix D(1f)	7 Dec 65	2748.1/815	8 Dec 1965
402c	Nozzle	D-30-C	11 Aug 65, Appendix D(1f)	10 Jan 66	2748.1/860	14 Feb 1966
403a	Nozzle	SG7-40-C	11 Aug 65, Appendix D(1g)	10 Jan 66	2748.1/860	14 Feb 1966
406	Nozzle	PP(412)-40-C	11 Aug 65, Appendix D(1j)	17 Sept 65	2748.1/765	23 Sept 1965
410	Nozzle	PH9-40-C	11 Aug 65, Appendix D(1n)	13 Oct 65	2748.1/785	26 Oct 1965
413	Pellet	D-35-BN	7 Jul 65 (4)	27 Dec 65	2748.1/849	24 Jan 1966
414	Supersonic Pipe	N151-35-C	28 Oct 65, Appendix A(1g)	24 Nov 65	2748.1/815	8 Dec 1965
415	Supersonic Pipe	PP(412)-35-GU	28 Oct 65, Appendix A(1e)	16 Nov 65	2748.1/815	8 Dec 1965
416	Supersonic Pipe	PPP-35-GU	28 Oct 65, Appendix A(1f)	12 Nov 65	2748.1/815	8 Dec 1965
417	Supersonic Pipe	DN-35-GU	28 Oct 65, Appendix A(1b)	12 Nov 65	2748.1/815	8 Dec 1965
418[3]	Supersonic Pipe	9-35-GU	28 Oct 65, Appendix A(1a)	12 Nov 65	2748.1/815	8 Dec 1965

TABLE 7 (CONTINUED)
TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Code	RTD Letter Reference	Date of Shipment	Detailed Letter Report	
					Reference Number	Date
419[3]	Supersonic Pipe	9-35-GU	28 Oct 65, Appendix A(1a)	12 Nov 65	2748.1/815	8 Dec 1965
420	Supersonic Pipe	SG7-35-GU	28 Oct 65, Appendix A(1c)	12 Nov 65	2748.1/815	8 Dec 1965
421	Supersonic Pipe	9-35-GC	28 Oct 65, Appendix A(1d)	16 Nov 65	2748.1/815	8 Dec 1965
422	Pellet	9-35-QF	28 Oct 65, (4)	27 Dec 65	2748.1/849	24 Jan 1966
423	Nozzle	PPF-40-C	28 Oct 65, (5a)	10 Jan 66	2748.1/860	14 Feb 1966
424a	Nozzle	PPF-40-R	28 Oct 65, (5b)	10 Jan 66	2748.1/860	14 Feb 1966
424b	Nozzle	PPF-40-R	28 Oct 65, (5b)	26 Jan 66	2748.1/860	14 Feb 1966
425	Supersonic Pipe	9-35-C	Telecon P. Pirrung 15 Nov 65	7 Dec 65	2748.1/815	8 Dec 1965
451/437	Hot Gas Flow	PPP-35-GU	1 Feb 66, Appendix A(1d)	14 Feb 66	2748.1/860	14 Feb 1966
452/437	Lam. Square	PPP-35-GU	1 Feb 66, Appendix A(1d)	14 Feb 66	2748.1/860	14 Feb 1966
453/437	Pellet	PPP-35-GU	1 Feb 66, Appendix A(2a)	14 Feb 66	2748.1/860	14 Feb 1966
457/441	Hot Gas Flow	9-35-GU	1 Feb 66, Appendix A(2a)	14 Feb 66	2748.1/860	14 Feb 1966
458/441	Lam. Square	9-35-GU	1 Feb 66, Appendix A(2a)	14 Feb 66	2748.1/860	14 Feb 1966
459/441	Pellet	9-35-GU	1 Feb 66, Appendix A(2a)	14 Feb 66	2748.1/860	14 Feb 1966

1 Replacement for specimens shipped on Data Sheet No. 338.

2 Replacement for specimens shipped on Data Sheet No. 395a.

3 Two parts were individually molded. Sixteen parts were machined from one laminate.

TABLE 8

TEST SPECIMENS LISTED ACCORDING TO TYPE OF REINFORCEMENT

Type of Reinforcement	Type of Resin		Data Sheet Number
Asbestos	PNPII		387
Boron Fibers	DEN 438		396
Boron Nitride Fibers	DEN 438		413
Carbon Cloth CCA-1	91LD		353 357 383 395a 395b 399a 399b 425
	91LD & Polyphenylene (Abchar 700)		370
	Chrome-P		375
	DEN 438		402a 402b 402c
	Imidite 1850 Resin System	Specimens impregnated with DEN 438 resin	346 347 348
		Specimens impregnated with QZ8-0903 resin	349 350 351
		Specimens impregnated with R-7144 resin	343 344 345
		Specimens impregnated with Sylgard 182 resin	340 341 342
	Imidite 2803 (AFR 151) Resin System		392 414
	PH990		410
	Polyphenylene (Abchar 412)		406
	Polyphenylene (Abchar 413)		369 372

Type of Reinforcement	Type of Resin	Data Sheet Number
Carbon Cloth CCA-1	2, 2-bis (p-hydroxyphenyl)-Propane phenol formaldehyde	423
	Skygard 700	403a
Carbon Cloth CCA-1 - Low Alkalinity (SS-1641)	91LD	359 360
Carbon Silica Fabric	91LD	384 385
Fused Silica Fabric (coated with Pyrolytic Carbon)	91LD	390
Glass Cloth, Style 181, A1100 Finish	Imidite 2803 Resin System (AFR-151)	356
	Polyphenylene (Abchar 413)	368
Graphite Cloth G1550 (uncoated)	91LD	400 418 419 457/441 458/441 459/441
	2, 7 Dihydroxynaphthalene phenol formaldehyde	417
	Polyphenylene (Abchar 412)	415
	Phenylphenol phenol formaldehyde	416 451/437 452/437 453/437
	Skygard 700	420
Graphite Cloth G1550 (Pyrolytic Graphite coated 1μ thick)	91LD	421
Graphite Cloth G1550 (Pyrolytic Graphite-Boron Alloy coated 1μ thick)	91LD	330
Graphite Cloth G1550 (Pyrolytic Graphite-Boron Alloy coated 2.5μ thick)	91LD	376
Polybenzimidazole Fibers	91LD	235 236

TABLE 8 (CONTINUED)

TEST SPECIMENS LISTED ACCORDING TO TYPE OF REINFORCEMENT

Type of Reinforcement	Type of Resin	Data Sheet Number
Quartz Fabric 581	91LD	422
Rayon Fabric	91LD	336 337
Rayon Silica Fabric Rayon Silica Fabric	91LD 91LD	338 339 389
Refrasil Cloth C100-48	91LD	354 358 401
	Chrome-P	373 374
	DEN 438	386
	Imidite 1850 Resin System	173

Type of Reinforcement	Type of Resin	Data Sheet Number
Refrasil Cloth C100-48 (continued)	Imidite 2803 Resin System (AFR-151)	364 393
	PNPII	388
	2, 2-bis (p-hydroxyphenyl) - Propane phenol formaldehyde	424a 424b
Refrasil Cloth and Carbon Black 452-00156 as filler	Polyphenylene Sulfide	352
Sapphire Wool Fibers (1-3μ dia.)	91LD	331
SC-70 Silicon Carbide Fibers and Bulk Crystal Mix (1-5μ dia.)	91LD	334
Silicon Carbide Wool Fibers (2-8μdia.	91LD	333
T-70 Fiber Crystal (Mixture of Aluminum Nitride and Aluminum Oxide Fibers	91LD	335

TABLE 9

TEST SPECIMENS LISTED ACCORDING TO TYPE OF RESIN

Tradename or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number
91 LD	Phenolic	Carbon Cloth CCA-1	353 357 383 395a 395b 399a 399b 425
		Carbon Cloth CCA-1 Low Alkalinity (SS-1641)	359 360
		Carbon Silica Fabric	384 385
		Fused Silica Fabric (coated with Pyrolytic carbon)	390
		Graphite Cloth G1550 (uncoated)	400 418 [illegible] 457 [illegible] 458 441 459/441
		Graphite Cloth G1550 (Pyrolytic Graphite coated 1μ thick)	421
		Graphite Cloth G1550 (Pyrolytic Graphite-Boron Alloy coated 1μ thick)	330
		Graphite Cloth G1550 (Pyrolytic Graphite-Boron Alloy coated 2.5μ thick)	376
		Polybenzimidazole Fibers	235 236
		Quartz Fabric	422

Tradename or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number
91LD	Phenolic	Rayon Fabric	336 337
		Rayon Silica Fabric	338 339 389
		Refrasil Cloth C100-48	354 358 401
		Sapphire Wool Fibers (1-3μ dia)	331
		SC-70 Silicon Carbide Fibers and Bulk Crystal Mix (1-5μ dia)	334
		Silicon Carbide Wool Fibers (2-8μ dia)	333
		T-70 Fiber Crystal (Mixture of Aluminum Nitride and Aluminum Oxide Fibers	335
91LD and Abchar 700	Phenolic and insoluble branched Polyphenylene	Carbon Cloth CCA-1	370
Chrome P	Chrome based metal organic phenolic	Refrasil Cloth C100-48	373 374
		Carbon Cloth CCA-1	375
DEN 438	Epoxy	Boron Fibers	396
		Boron Nitride Fibers	413
		Carbon Cloth CCA-1	402a 402b 402c
		Refrasil Cloth C100-48	386
2, 7 Dihydroxy-naphthalene phenol formaldehyde	Modified Phenolic	Graphite Cloth G1550 (uncoated)	417

TABLE 9 (CONTINUED)

TEST SPECIMENS LISTED ACCORDING TO TYPE OF RESIN

Tradename or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number
Imidite 1850 Resin System	Polybenzimidazole (Phenol Blocked)	Refrasil Cloth C100-48	173
Imidite 1850 Resin System and DEN 438 [1]	Polybenzimidazole and Epoxy	Carbon Cloth CCA-1	346 347 348
Imidite 1850 Resin System and QZ8-0903 [1]	Polybenzimidazole and Silicone-Epoxy	Carbon Cloth CCA-1	349 350 351
Imidite 1850 Resin System and R-7146 [1]	Polybenzimidazole and Silicone	Carbon Cloth CCA-1	343 344 345
Imidite 1850 Resin System and Sylgard 182 [1]	Polybenzimidazole and Silicone	Carbon Cloth CCA-1	340 341 342
Imidite 2803 Resin System (AFR-151)	Polybenzimidazole (Amide Blocked)	Carbon Cloth CCA-1	392 414
		Glass Cloth, Style 181, A1100 Finish	356
		Refrasil Cloth C100-48	364 393
PH 990	Phosphonitrilic-Modified Phenolic	Carbon Cloth CCA-1	410

Tradename or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number
Phenylphenol phenol formaldehyde	Modified Phenolic	Graphite Cloth G1550 (uncoated)	416 451/437 452/437 453/437
PNPII	Carborane	Asbestos	387
		Refrasil Cloth C100-48	388
Polyphenylene (Abchar 412)	m-Polyphenylene	Carbon Cloth CCA-1	406
		Graphite Cloth G1550 (uncoated)	415
Polyphenylene (Abchar 413)	m-Polyphenylene	Carbon Cloth CCA-1	369 372
		Glass Cloth, Style 181, A1100 Finish	368
Polyphenylene Sulfide	Polyphenylene Sulfide	Refrasil Cloth C100-48 and Carbon Black 452-00156 as Filler	352
2, 2-bis (p-hydroxyphenyl) - Propane phenol formaldehyde	Modified Phenolic	Carbon Cloth CCA-1	423
		Refrasil Cloth C100-48	424a 424b
Skygard 700	Polyamide-imide	Carbon Cloth CCA-1	403a
		Graphite Cloth G1550 (uncoated)	420

1 Machined Imidite 1850 resin system-carbon cloth CCA-1 specimens were impregnated with resin under pressure.

TABLE 10

FABRICATION DETAILS - HOT GAS FLOW SPECIMENS

Data Sheet Number	Type of Specimen[1]	Dimensions of Molded or Laminated Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
						Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
353	Type D	18" x 12" x 1/2"	9-40-C	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	200	240	B-1
354	Type D	12" x 9" x 1/2"	9-40-R	1/0.25 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	200	240	B-1
357-1 357-2	Type A	3-1/2" x 1-7/8" x 1-1/2"	9-40-C	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	5,000	120	B-1
358-1 358-2	Type A	3-1/2" x 1-7/8" x 1-1/2"	9-40-R	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	5,000	120	B-1
383	Type A	3-1/2" x 1-7/8" x 1-1/2"	9-40-C	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	5,000	120	B-1
395a-1 395a-2	Type E	3-1/2" x 1-7/8" x 1-1/2"	9-40-C	1/0.5 Acetone	Spatula Coating	-	60 min at 160°F	30 min at 225°F	300	5,000	120	B-1
395b-1 395b-2 395b-3	Type E	3-1/2" x 1-7/8" x 1-1/2"	9-40-C	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F[2]	200 and 300	10,000	60-60 45-60 45-60	B-1
451/437[3]	Type D	3-1/3" x 2" x 1/2"	PPP-35-GU	Used as received	Spatula Coating	960	30 min at 160°F	15 min at 240°F	300	300	120	B-1
451/441[4]	Type D	3-1/3" x 2" x 1/2"	9-35-GU	1/0.7 Acetone	Spatula Coating	120	60 min at 150°F	30 min at 225°F	300	180	180	B-1

One postcure schedule was used:

B-1 18 hrs at 275°F, 72 hrs from 275° to 400°F, 4 hrs at 400°F, 7 hrs cooling to below 200°F.

1 Type A - 3-1/3" x 1-3/4" x 1/2" (ea ply 1-3/4" x 1/2"). Two parts were machined from each 3-1/2" x 1-7/8" x 1-1/2" blank molded in the hot gas flow mold.
Type D - 3-1/3" x 2" x 1/2" (ea ply 3-1/3" x 2"). Parts were machined from 1/2" thick laminates.
Type E are molded from impregnated 3/8" squares of reinforcement in the hot gas flow mold. Two parts 3-1/3" x 1-3/4" x 1/2" are machined from each blank.

2 Preform was vacuum dried for 16 hours after "B" staging.

3 Specimens machined from laminate listed on Data Sheet No. 437.

4 Specimens machined from laminate listed on Data Sheet No. 441.

TABLE 11
FABRICATION DETAILS - LAMINATES

Data Sheet Number	Dimensions of Molded Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
332[1]	16" x 12" x 5/8"	N-35-C	Not applicable	Prepreg purchased as Imidite 4834	N. A.	N. A.	N. A.	700	200	180	H-2
368	6" x 6" x 1/8"	PP413-35-G	Used as received	Dip coating	20	30 min at 160°F	None	400	300	120	I-1
437	9" x 6-3/4" x 1/2"	PPP-35-GU	Used as received	Spatula Coating	960	20 min at 160°F	15 min at 240°F	300	300	120	B-1
441	18" x 17-1/2" x 1/2"	9-35-GU	1/0.7 Acetone	Spatula Coating	120	60 min at 160°F	30 min at 225°F	300	180	180	B-1

Three postcure schedules were used:

B-1 18 hours at 275°F, 7 hours from 275° to 400°F, 4 hours at 400°F, 7 hours cooling to below 200°F.

H-2 24 hours at 600°F, 24 hours at 650°F, 24 hours at 700°F, 24 hours at 750°F and 8 hours at 800°F. Parts were postcured in a helium atmosphere.

I-1 18 hrs at 275°F, 114 hrs from 275°F to 600°F, 7 hrs cooling to below 200°F. Part was postcured in a helium atmosphere.

1 The polybenzimidazole-carbon cloth laminate fabricated on Data Sheet No. 332 was machined into laminates and pellets and bonded and machined into rocket nozzles. Machined specimens are listed on Data Sheet Nos. 340, 341, 342, 343, 344, 345, 346, 347, 348, 349, 350 and 351.

TABLE 12

FABRICATION DETAILS - LAMINATED SQUARES

Data Sheet Number	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
				Air Dry Min	Oven Dry		Temp, °F	Pressure, PSI	Time, Min	
341[1] L-SY-1 L-SY-2	N-35-C(SY)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150° F 60 min at 212° F 30 min at 300° F	N.A.	N.A.	N.A.	N.A.	N.A.
344[1] L-S-1 L-S-2	N-35-C(S)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150° F 60 min at 212° F 30 min at 300° F	N.A.	N.A.	N.A.	N.A.	N.A.
347[1] L-D-1 L-D-2	N-35-C(D)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150° F 60 min at 212° F 30 min at 300° F	N.A.	N.A.	N.A.	N.A.	N.A.
350[1] L-SE-1 L-SE-2	N-35-C(SE)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150° F 60 min at 212° F 30 min at 300° F	N.A.	N.A.	N.A.	N.A.	N.A.
452/437[2]	PPP-35-GU	Used as received	Spatula coating	960	20 min at 160° F	15 min at 240° F	300	300	120	B-1
458/441[3]	9-35-GU	1/0.7 Acetone	Spatula coating	120	60 min at 160° F	30 min at 225° F	300	180	180	B-1

One postcure schedule was used:

B-1 18 hrs at 275° F, 72 hrs from 275° to 400° F, 4 hrs at 400° F, 7 hrs cooling to below 200° F.

1 Part of the polybenzimidazole-carbon cloth laminate fabricated on Data Sheet No. 332 was machined into laminated squares. Information listed under "Type of Impregnation" and "Drying Conditions" refers to the method of impregnating the machined specimens and to the resin cure cycle.

2 Specimens machined from laminate listed on Data Sheet No. 437.

3 Specimens machined from laminate listed on Data Sheet No. 441.

TABLE 13

FABRICATION DETAILS - PELLET SPECIMENS

Data Sheet Number	Dimensions of Molded Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min.	
235-1	3/4" dia x 1/2"	9-45-PBIF	1/2.4 Acetone	Buchner Funnel	-	30 min at 160°F	45 min at 220°F	300	3,300	60	None
235-2	3/4" dia x 1/2"	9-45-PBIF	1/2.4 Acetone	Buchner Funnel	-	30 min at 160°F	45 min at 220°F	300	3,300	60	None
235-3	3/4" dia x 1/2"	9-45-PBIF	1/2.4 Acetone	Buchner Funnel	-	30 min at 160°F	45 min at 220°F	300	3,300	60	None
236	3/4" dia x 1/2"	9-35-PBIF	1/3.3 Acetone	Buchner Funnel	-	30 min at 160°F	20 min at 225°F	300	3,300	60	None
330	3-1/2" dia x 1/2" [1]	9-35-GCB	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	3,300	120	B-1
331-1 331-2	3/4" dia x 1/2"	9-35-SW	1/32 Acetone 1/30 Acetone	Soaking	60	60 min at 160°F	30 min at 225°F	300	3,300	120	B-1
333-1 333-2	3/4" dia x 1/2"	9-35-SCW	1/40 Acetone	Soaking	60	60 min at 160°F	25 min at 225°F	300	3,300	120	B-1
334-1 334-2	3/4" dia x 1/2"	9-35-SC70	1/5 Acetone	Soaking	120	240 min at 160°F	45 min at 225°F	300	3,300	120	B-1
335-1 335-2	3/4" dia x 1/2"	9-35-T70	1/2.5 Acetone	Soaking	120	240 min at 160°F	35 min at 225°F	300	3,300	120	B-1
336	2" dia x 1/2" [1]	9-35-RF	1/0.9 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	3,300	120	B-1
338	2" dia x 1/2" [1]	9-35-RSF	1/0.75 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	3,300	120	B-1 [2]
342/332 [3]	3/4" dia x 1/2"	N-35-C(SY)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N.A.	N.A.	N.A.	N.A.	N.A.

TABLE 13 (CONTINUED)
FABRICATION DETAILS - PELLET SPECIMENS

Data Sheet Number	Dimensions of Molded Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min.	
345/332$_3$	3/4" dia x 1/2"	N-35-C(S)	N. A.	Vacuum followed by high pressure	N. A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N. A.	N. A.	N. A.	N. A	N. A.
348/332$_3$	3/4" dia x 1/2"	N-35-C(D)	N. A.	Vacuum followed by high pressure	N. A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N. A.	N. A.	N. A.	N. A.	N. A.
351/332$_3$	3/4" dia x 1/2"	N-35-C(SE)	N. A.	Vacuum followed by high pressure	N. A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N. A.	N. A.	N. A.	N. A.	N. A.
352-1 352-2 352-3	3/4" dia x 1/2"	FS-35-RCB	N. A.	Dry powder layup	N. A.	N. A.	N. A.	600	22, 600	60	B-1
360	2" dia x 1/2"$_1$	9-35-CLA	1/0. 5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	3, 300	120	B-1
369	3-1/2" dia x 1/2"$_1$	PP413-35-C	Used as received	Dip coating	20	20 min at 160°F	None	400	3, 000	120	I-1
370	2" dia x 1/2"$_1$	9PP-45-C	1/0. 13 Acetone	Spatula Coating	20	35 min at 230°F	None	325	3, 000	120	B-1
374	2" dia x 1/2"$_1$	CP-35-R	1/0. 25 Acetone	Spatula Coating	120	60 min at 180°F	-	180 250	3, 300	120 300	J-1
375	2" dia x 1/2"$_1$	CP-35-C	1/0. 3 Acetone	Spatula Coating	120	60 min at 180°F	-	180 250	3, 300	120 300	J-1

TABLE 13 (CONTINUED)
FABRICATION DETAILS - PELLET SPECIMENS

Data Sheet Number	Dimensions of Molded Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min.	
376	3-1/2" dia x 1/2"[1]	9-35-GCR (2.5)	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	3,300	120	B-1
385	2" dia x 1/2"[1]	9-35-CSF	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	25 min at 225°F	300	3,300	120	B-1
387	1-5/8" dia x 2-1/2"[4]	PNP II-40-A	Not applicable	Received as RM 9002	-	-	-	700	6,000	240	K
388	1-5/8" dia x 2-1/2"[4]	PNP II-40-R	Not applicable	Dry Powder Layup	-	-	-	700 800	[0][5]	60 60	K
389	2" dia x 1/2"[1]	9-35-RSF[6]	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	3,300	120	B-1
390-1 390-2	3/4" dia x 1/2"	9-35-FSF (PC)	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	25 min at 225°F	300	3,300	120	B-1
396-1 396-2 396-3	1" x 1" x 1/2"[1]	D-30-BF	Not applicable	Impregnated by AFML	-	-	-	300	200	120	B-1
413	3/4" dia x 1/2"	D-35-BN	1/34 Acetone	Soaking	180 min Vacuum Dry	60 min at 160°F	10 min at 225°F	300	22,600	120	B-1
422	2" dia x 1/2"[1]	9-35-QF	1/1 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	3,300	120	B-1

TABLE 13 (CONTINUED)
FABRICATION DETAILS - PELLET SPECIMENS

Data Sheet Number	Dimensions of Molded Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min.	
453/437[7]	3/4" dia x 1/2"	PPP-35-GU	Used as received	Spatula Coating	960	20 min at 160°F	15 min at 240°F	300	300	120	B-1
459/441[8]	3/4" dia x 1/2"	9-35-GU	1/0.7 Acetone	Spatula Coating	120	60 min at 160°F	30 min at 225°F	300	180	180	B-1

Four postcure schedules were used:

B-1 18 hrs at 275°F, 72 hrs from 275° to 400°F, 4 hrs at 400°F, 7 hrs cooling to below 200°F.[9]

I-1 18 hrs at 275°F, 114 hrs from 275° to 600°F, 6 hrs cooling to below 200°F. Parts were postcured in a helium atmosphere.

J-1 One (1) hr each at 150°F, 200°F, 250°F, 300°F, 350°F, 2 hrs at 400°F, 7 hrs cooling to below 200°F.

K After machining off the surface skin, specimen was placed in a room temperature oven. The temperature was raised to 250°F over 16 hours, then held at 250°F for 12 hrs. The temperature was then raised at the rate of 50°F per 12 hrs until 800°F. After being held at 800°F for 24 hrs, the part was cooled to below 200°F before removing.

1 Specimens prepared in molds other than the 3/4" disc mold were machined to 3/4" diameter x 1/2" thick pellets.

2 During postcure, the balancing motor, on a temperature controller burned out. The disc was subjected to 500°F for an unknown length of time.

3 Part of the polybenzimidazole-carbon cloth laminate fabricated on Data Sheet No. 332 was machined into pellets. Information listed under "Type of Impregnation" and "Drying Conditions" refers to the method of impregnating the machined specimens and to the resin cure cycle.

4 Pellets were machined from rocket nozzle segments which fractured when removed from the mold.

5 Excessive resin flow resulted in the punch landing on the mold, followed by loss of pressure on the part.

6 These parts are replacements for specimens shipped on Data Sheet No. 338.

7 Specimens machined from laminate listed on Data Sheet No. 437.

8 Specimens machined from laminate listed on Data Sheet No. 441.

9 Specimens on Data Sheet No. 370 were postcured in helium. Specimens on Data Sheet No. 396 were placed in a room temperature oven. The oven temperature was raised to 275°F over 16 hours.

TABLE 14
FABRICATION DETAILS - ROCKET NOZZLES

Data Sheet Number	Nozzle Number	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
173	K-1-1	N-35-R	Used as received	Impregnated cloth (received from AFML) + Dry powder layup	-	-	-	600 700	5,000	180	H-3
337	K-38	9-40-RF	1/1 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
339	K-39-1	9-40-RSF	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
340	K-40-1	N-35-C(SY)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N.A.	700	5,000	180	N.A.
343	K-41-1	N-35-C(S)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N.A.	700	5,000	180	N.A.
346	K-42-1	N-35-C(D)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N.A.	700	5,000	180	N.A.
349	K-43-1	N-35-C(SE)	N.A.	Vacuum followed by high pressure	N.A.	240 min at 150°F 60 min at 212°F 30 min at 300°F	N.A.	700	5,000	180	N.A.

TABLE 14 (CONTINUED)

FABRICATION DETAILS - ROCKET NOZZLES

Data Sheet Number	Nozzle Number	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
356	K-45	N151-30-G	N. A.	Dry powder layup	N. A.	N. A.	N. A.	700	3,000	180	H-1
359	K-46-1 K-46-2	9-40-CLA	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
364	K-44	N151-40-R	N. A.	Dry powder layup	N. A.	N. A.	N. A.	700	10,000	180	$H\text{-}1_4$
372	K-51-1 K-51-2	PP(413)-40-C	Used as received	Dip coating	20	20 min at 160°F	-	400	10,000	120	I-3
373	K-48-1	CP-40-R	1/0.2 Acetone	Spatula Coating	120	60 min at 180°F	-	180 250	10,000	120 300	J-2
384	K-49-1 K-49-2	9-40-CSF	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
386	K-50	D-40-R	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	60 min at 225°F	300	10,000	960	B-1
392	K-52-1 K-52-2	N151-40-C	Used as received	Preimpregnated by Narmco	-	-	-	600-700	10,000	60-120	H-1
393	K-53-1 K-53-2	N151-40-R	Not applicable	Preimpregnated by Narmco + dry powder layup	-	-	-	700	10,000	180	H-1
399a	K-47-1 K-47-2	9-40-C	1/0.5 Acetone	Spatula Coating	-	60 min at 160°F	20 min at 225°F	300_5 300	10,000	60 120	B-1
399b	K-47-3	9-40-C	1/0.5 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	10,000	120	B-1
400	K-58-1 K-58-2	9-40-GU	1/0.4 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	10,000	120	B-1
401	K-59-1 K-59-2 K-59-3	9-40-R	1/0.3 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	10,000	120	B-1

TABLE 14 (CONTINUED)

FABRICATION DETAILS - ROCKET NOZZLES

Data Sheet Number	Nozzle Number	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
402a	K-60-1 K-60-2	D-30-C	1/1.5 Acetone	Spatula Coating	30	60 min at 160°F	20 min at 225°F	300	10,000	120	B-3
402b	Special Cylindrical Blank[6]	D-30-C	1/0.7 Acetone	Spatula Coating	30	60 min at 160°F	20 min at 225°F	300	10,000	120	B-3
402c	K-60-3	D-30-C	1/5 Acetone	Spatula Coating	30	60 min at 160°F	20 min at 220°F	300	15,000	120	B-3
403a	K-61-1	SG7-40-C	1/0.3 Acetone	Spatula Coating	960	60 min at 200°F	30 min at 250°F[7]	500	2,000	180	E
406	K-64-1 K-64-2	PP(412)-40-C	Used as received	Spatula Coating	20	10 min at 160°F	-	380	10,000	120	I-2
410	K-68-1 K-68-2	PH9-40-C	1/2 Acetone	Spatula Coating	20	60 min at 160°F	10 min at 200°F	450	10,000	60	B-2
423	K-71-1 K-71-2	PPF-40-C	Used as received	Spatula Coating	20	20 min at 160°F	10 min at 220°F	300	10,000	120	B-1
424a	K-72-1	PPF-40-R	Used as received	Spatula Coating	20	20 min at 160°F	10 min at 220°F	300	10,000	120	B-1
424b	K-72-2	PPF-40-R	Used as received	Spatula Coating	20	20 min at 160°F	10 min at 220°F	300	10,000	120	B-1

TABLE 14 (CONTINUED)

FABRICATION DETAILS - ROCKET NOZZLES

Nine postcure schedules were used:

B-1	18 hrs at 275°F, 72 hrs from 275° to 400°F, 4 hrs at 400°F, 7 hrs cooling to below 200°F.
B-2	18 hrs at 275°F, 72 hrs from 275° to 400°F, 4 hrs at 400°F, 4 hrs at 425°F, 7 hrs cooling to below 200°F.
B-3	17 hrs at 275°F, 6 hrs from 275° to 400°F, 1 hr at 400°F, 7 hrs cooling to below 200°F.
E	24 hrs at 375°F, 24 hrs at 435°F, 24 hrs at 475°F, 24 hrs at 575°F (6 hrs between temp.) 7 hrs cooling to below 200°F.
H-1	24 hrs at 600°F, 24 hrs at 650°F, 24 hrs at 700°F, 6 hrs at 750°F, 6 hrs at 800°F, 6 hrs at 850°F. Cooled to below 200°F. Parts postcured in argon atmosphere.
H-3	24 hrs at 600°F, 24 hrs at 650°F, 24 hrs at 700°F, 24 hrs at 750°F, 8 hrs at 800°F and 3 hrs at 700°F. Part postcured in nitrogen atmosphere.
I-2	18 hrs at 275°F, 72 hrs from 275° to 400°F, 39 hrs from 400° to 550°F, 7 hrs cooling to below 200°F. Parts were postcured under helium atmosphere.
I-3	18 hrs at 275°F, 108 hrs from 275°F to 550°F, 6 hrs at 550°F, 7 hrs cooling to below 200°F. Parts postcured in helium atmosphere.
J-2	Three (3) hrs each at 150°, 200°, 250°, 300°, 350°F, 6 hrs at 400°F, 7 hrs cooling to below 200°F.

1 Material placed in 600°F mold. Temperature increased to 700°F within 10 minutes.

2 During postcure, the balancing motor on a temperature controller burned out. The nozzle blank was subjected to 500°F for an unknown length of time.

3 Part of the polybenzimidazole-carbon cloth laminate fabricated on Data Sheet No. 332 was bonded together and machined into rocket nozzles. Information listed under "Type of Impregnation," and "Drying Conditions" refers to the method of impregnating the machined specimens and to the resin cure cycle. Information listed under "Molding Conditions" refers to bonding 2" x 2" x 1/2" pieces to form a rocket nozzle blank.

4 Because of a failure of a temperature controller, part remained at 700°F for 48 hours, at 800°F for 12 hours and at 850°F for 12 hours.

5 Held at contact pressure for 60 minutes at 200°F.

6 This specimen was a rocket nozzle blank whose resin content was 1.3 wt-% over the allowable tolerance. The unmachined cylindrical blank was shipped as an extra specimen.

7 Prepreg was vacuum dried for 16 hours after "B" staging.

TABLE 15

FABRICATION DETAILS - SUPERSONIC PIPE SPECIMENS

Data Sheet Number	Dimensions of Molded Specimen	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
414	5" x 3" x 1.5"[1]	N151-35-C	N.A.	Preimpregnated by Narmco	-	-	-	700	500	180	H-1[2]
415-1 415-2	1.675" dia x 1.5"	PP(412)-35-GU	Used as received	Dip coated	30	60 min at 160°F	-	420	10,000	120	I-3
416-1 416-2	1.675" dia x 1.5"	PPP-35-GU	1/0.7 Acetone	Spatula Coating	-	20 min at 160°F	15 min at 240°F	300	10,000	120	B-1
417-1 417-2	1.675" dia x 1.5"	DN-35-GU	1/1 Acetone	Spatula Coating	30	15 min at 160°F	15 min at 230°F	300	10,000	120	B-1
418-1 418-2	1.675" dia x 1.5"	9-35-GU	1/0.7 Acetone	Laboratory Coater[3]	Tower temperature - 240°F Speed of cloth, Run #1-12 in./min Speed of cloth, Run #2-16 in./min			300	10,000	120	B-1
419	9" x 8-1/2" x 1.5"[1]	9-35-GU	1/0.7 Acetone	Laboratory Coater[3]	Tower temperature - 240°F Speed of cloth, Run #1-12 in./min Speed of cloth, Run #2-16 in./min			300	400	240	B-1
420	5" x 3" x 1.5"[1]	SG7-35-GU	1/0.4 Acetone	Spatula Coating	35	60 min at 200°F	30 min at 250°F	600	500	120	E
421-1 421-2	1.675" dia x 1.5"	9-35-GC	1/0.6 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	10,000	120	B-1
425	5" x 3" x 1.5"[1]	9-35-C	1/0.7 Acetone	Spatula Coating	60	60 min at 160°F	20 min at 225°F	300	500	120	B-1

Four postcure schedules were used:

B-1 18 hrs at 275°F, 72 hrs from 275° to 400°F, 4 hours at 400°F, 7 hrs cooling to below 206°F.

E 24 hrs at 375°F, 24 hrs at 435°F, 24 hrs at 475°F, 24 hrs at 575°F (6 hrs between temp) 7 hrs cooling to below 200°F.

H-1 24 hrs at 600°F, 24 hrs at 650°F, 24 hrs at 700°F, 6 hrs at 750°F, 6 hrs at 800°F, 6 hrs at 850°F. Cooled to below 200°F. Parts postcured in argon atmosphere.

I-3 18 hrs at 275°F, 92 hrs from 275° to 550°F, 6 hrs at 550°F, 7 hrs cooling to below 200°F. Parts postcured in argon atmosphere.

1 1.675" diameter supersonic pipe specimens were machined from these blocks, plies being perpendicular to the vertical axis of the cylinders. The height of the cylinders was the thickness of the molded laminate.

2 The Imidite postcure schedule calls for 6 hrs at 800°F. The specimens were at that temperature when the programming cam stuck. They remained for 24 hrs at 800°F before the breakdown in the temperature schedule was discovered.

3 An equal number of plies from Runs 1 and 2 were combined a[illegible]ized

TABLE 16

MATERIAL SOURCES

Tradename or Designation	Type of Material	Source
91LD	Resin	American Reinforced Sales
Asbestos	Reinforcement	Raybestos-Manhattan
Boron Fibers	Reinforcement	AFML
Boron Nitride Fibers	Reinforcement	AFML
Carbon Black 452-00156	Filler	Godfrey Cabot Co.
Carbon Cloth CCA-1	Reinforcement	HITCO
Carbon Cloth CCA-1 - Low Alkalinity (SS-1641)	Reinforcement	HITCO
Carbon Silica Fabric	Reinforcement	AFML
Chrome-P	Resin	Thermo Resist
DEN 438	Resin	Thalco
2, 7 Dihydroxynaphthalene phenol formaldehyde	Resin	Hughes Aircraft
Fused Silica Fabric (coated with pyrolytic carbon)	Reinforcement	AFML
Glass Cloth, Style 181, A1100 Finish	Reinforcement	Thalco
Graphite Cloth G1550 (uncoated)	Reinforcement	HITCO
Graphite Cloth G1550 (Pyrolytic Graphite coated 1 μ thick)	Reinforcement	AFML
Graphite Cloth G1550 (Pyrolytic Graphite - Boron Alloy coated 1μ thick)	Reinforcement	AFML
Graphite Cloth G1550 (Pyrolytic Graphite - Boron Alloy coated 2.5μ thick)	Reinforcement	AFML
Imidite 1850 System	Resin	Narmco Materials
Imidite 2803 System (AFR-151)	Resin	Narmco Materials
PH990	Resin	El Monte Chemical
Phenylphenol phenol formaldehyde	Resin	Hughes Aircraft
PNPII	Resin	Olin Mathieson
Polybenzimidazole Fibers	Reinforcement	AFML
Polyphenylene (Abchar 412)	Resin	Hughes Aircraft
Polyphenylene (Abchar 413)	Resin	Hughes Aircraft
Polyphenylene (Abchar 700)	Resin	Hughes Aircraft
Polyphenylene Sulfide	Resin	AFML
2, 2-bis (p-hydroxyphenyl)-Propane phenol formaldehyde	Resin	Hughes Aircraft
Quartz Fabric 581	Reinforcement	J. P. Stevens
QZ8-0903	Resin	Dow Corning
R-7146	Resin	Dow Corning
Rayon Fabric	Reinforcement	AFML
Rayon Silica Fabric	Reinforcement	AFML
Refrasil Cloth C100-48	Reinforcement	HITCO
Sapphire Wool Fibers	Reinforcement	AFML
SC70 Silicon Carbide Fibers	Reinforcement	AFML
Silicon Carbide Wool Fibers	Reinforcement	AFML
*Skygard 700	Resin	Monsanto
Sylgard 182	Resin	Dow Corning
T-70 Fiber Crystals	Reinforcement	AFML

*Subsequently renamed Skybond 700 by manufacturer

Unclassified
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13. ABSTRACT

Precise processing techniques were used in preparing new ablative plastic composites. This research involved the use of novel heat resistant resins such as: cross-linked, branched polyphenylenes; para-polyphenylenes, an amide-blocked polybenzimidazole, a phosphonitrilic-modified phenolic, a carborane and a polyimide.

Novel materials used as reinforcements included boron fibers, boron nitride fibers, fused silica fabric coated with pyrolytic graphite, polybenzimidazole fibers, rayon-silica fabric, carbon-silica fabric, sapphire wool fibers and silicon carbide fibers.

Resin impregnation techniques used in preparing research specimens included spatula coating, dip coating, machine coating in a laboratory treater, Buchner funnel impregnation and dry powder layup.

Research specimens were prepared in various geometric configurations depending on their intended ablative characterization. Test specimen geometries included small pellets (3/4-inch diameter by 1/2-inch thick) for hyperthermal reentry screening, subscale rocket nozzles for solid- and liquid-propellant rocket motor screening and hot-gas flow specimens for channel-type hyperthermal tests.

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14 KEY WORDS	LINK A		LINK B		LINK C	
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Ablative Plastics						

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